

**PRELIMINARY EVALUATION OF SULPHUR SULPHIDIC
RESIDUE SEPARATIONS**



FINAL REPORT

by

J.R. Harlamovs

COMINCO LTD.

RESEARCH & DEVELOPMENT

TRAIL, B.C.

Work on the project was conducted under the auspices of
the Canada Centre for Mineral and Energy Technology,
Energy, Mines and Resources Canada.

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EXECUTIVE SUMMARY

The objective of the project was to examine the recovery of elemental sulphur from hydrometallurgical sulphur-sulphide residues. The project was to address the following general aims:

- (1) Obtain a literature survey of previous hydrometallurgical plant practices and of likely avenues for further study.
- (2) Determine what factors are important during the leaching of sulphides for the subsequent recoverability of elemental sulphur.
- (3) Examine the feasibility of physical and chemical sulphur recovery from secondary residues.
- (4) Compare various sulphur recovery routes.

The literature survey showed that many hydrometallurgical processes that treat metal sulphides and produce elemental sulphur as a by-product have been proposed but only a few have been put into practice. Very few of the proposed processes have addressed the actual problem of sulphur recovery.

In this report, the recoverability of elemental sulphur from primary leach residues and the factors that affect recoverability during leaching were left largely unaddressed. Lignosol® surfactant added during a ferric chloride leach of lead sulphide concentrates did not improve lead dissolution or the subsequent sulphur recovery by flotation.

The recovery of sulphur from Inco nickel sulphide anode slimes and Cominco's hot sulphur filter residue using physical separations such as cycloning and pelletization proved unsuccessful. The leaching of elemental sulphur from the two secondary residues anode slimes and hot sulphur filtration residue using carbitol (a glycol based solvent), showed that carbitol could be a suitable sulphur solvent. Water is completely miscible with carbitol, an advantage for handling wet cakes, but depresses the sulphur solubility.

Ammonium sulphite solution is a good lixiviant for sulphur, its disadvantage is that it is not recyclable. Sulphur, on dissolving in ammonium sulphite solution, produces an ammonium thiosulphate solution, which on purification could be used as a fertilizer.

Four lixiviants were compared for sulphur recovery from a primary residue; the residue was obtained from a ferric chloride leach of a lead concentrate. The four lixiviants examined were a lime-water slurry, carbitol, ammonium sulphite solution and ammonium sulphide solution. All four exhibited good properties for sulphur dissolution. Of the four lixiviants, carbitol produced the purest sulphur. Sparging hydrogen sulphide through the ammonium sulphite leach liquor prior to sulphur precipitation significantly upgraded the purity of the product sulphur.

In the future, the recovery of elemental sulphur from sulphidic hydrometallurgical residues will be important in financial and environmental terms. When recovering sulphur from metallurgical residues, each material and location will have its own unique problems and requirements. Consequently, it is difficult to compare the various methods of sulphur recovery realistically. The unexpected low availability of staff to carry out testwork and evaluation has compounded the problem of process comparison. Therefore, this project has been brought to an early conclusion without meeting all of its objectives. In the future, a particular residue at a specific operation should be targeted when evaluating different sulphur recovery processes. This will allow a realistic evaluation of the metallurgical requirements.

RÉSUMÉ EXÉCUTIF

L'objectif du project était d'examiner le recouvrement du soufre élémentaire des résidus de soufre-sulfide provenant des procédés hydrométallurgiques. Le project avait pour but les différents points suivants:

- (1) Obtenir une étude littéraire des procédés utilisés précédemment dans les usines hydrométallurgiques ainsi que des avenues possibles pour des études futures.
- (2) Déterminer les facteurs importants durant la lixiviation des sulfures pour le recouvrement subséquent du soufre élémentaire.
- (3) Examiner la possibilité de recouvrir physiquement et chimiquement le soufre provenant des résidus secondaires.
- (4) Comparer les différentes avenues pour le recouvrement du soufre.

L'étude littéraire a démontrée que plusieurs procédés hydrométallurgiques qui traitent les métaux sulfurés et qui produisent du soufre élémentaire comme sous-produit ont été proposés mais que seulement un petit nombre a été mis en pratique. Très peu des procédés proposés ont adressé le problème actuel du recouvrement du soufre.

Dans ce rapport, le recouvrement du soufre élémentaire des résidus provenant d'une lixiviation primaire et les facteurs qui affectent ce recouvrement durant la lixiviation ont été largement laissés de côté. Le surfactant Lignosol® ajouté durant la lixiviation au chlorure ferrique de concentrés de sulfures de plomb n'a pas amélioré ni la dissolution du plomb ni le recouvrement du soufre par flotation.

Le recouvrement du soufre des boues d'anodes de sulfure de nickel de la compagnie Inco ainsi que le résidu de sulfure de la filtration à chaud de la compagnie Cominco, n'a pas eu de succès en utilisant des techniques de séparations physiques tels que le cyclonage et le bouletage.

La lixiviation au carbitol (un solvant à base de glycol) du soufre élémentaire provenant des deux résidus secondaires mentionnés ci-dessus a démontré que le carbitol pourrait être un solvant

approprié pour le soufre. L'eau est complètement miscible avec le carbitol. Ceci donne un avantage pour la manipulation des gâteaux humides, mais par contre diminue la solubilité du soufre.

La solution des sulfites d'ammonium est un bon lixiviant pour le soufre; son désavantage est qu'elle ne peut être recyclée. Le soufre, lorsqu'il est dissout dans une solution de sulfite d'ammonium, produit une solution de thiosulphate d'ammonium laquelle peut être utilisée après purification comme fertilisant.

Quatre lixiviants furent comparés pour le recouvrement du soufre d'un résidu primaire provenant de la lixiviation en milieu ferrique d'un concentré de plomb. Les quatre lixiviants examinés furent une pulpe chaux-eau, le carbitol, une solution de sulfite d'ammonium et une solution de sulfure d'ammonium.

Tous les quatres ont présenté de bonnes propriétés pour la dissolution du soufre. De ces quatre lixiviants, le carbitol a produit le soufre le plus pur. Le fait de faire barboter du sulfure d'hydrogène à travers la liqueur de lixiviation au sulfite d'ammonium, préalablement à la précipitation du soufre, a augmenté de manière significative la pureté du soufre produit.

Dans le futur, le recouvrement du soufre élémentaire des résidus provenant de procédés hydrométallurgiques sera important tant au point financier qu'environnemental. Lors du recouvrement du soufre provenant de résidus métallurgiques, chaque matériel et chaque emplacement auront leurs propres problèmes et exigences. Conséquemment il est difficile de comparer de façon réaliste les différentes méthodes de recouvrement du soufre. La faible disponibilité inattendue du personnel afin de poursuivre les essais et l'évaluation a compromis le problème de la comparaison du procédé. Par conséquent, ce projet fut amené à une conclusion anticipé sans avoir rencontré tous ses objectifs. Dans le futur, un résidu particulier à une opération spécifique devrait être ciblé lors de l'évaluation des différents procédés de recouvrement du soufre. Ceci permettra une évaluation réaliste des exigences métallurgiques.

INTRODUCTION

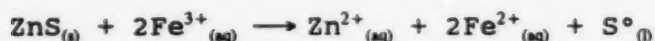
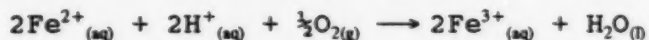
Sulphur is a by-product from both the metallurgical and petrochemical industries. In the petrochemical industry, hydrogen sulphide is recovered from sour gas and is converted to elemental sulphur. Various processes exist and the chemistry of the sulphur recovery technology is well established. In comparison, the recovery of sulphur from metallurgical operations is somewhat limited. In the majority of processes, sulphur is recovered as sulphuric acid, sulphate in fertilizer or sulphates in waste residues. Environmental pressures are making the production of sulphuric acid or the disposal of waste sulphate residues unattractive. In the last twenty years a large number of hydrometallurgical processes that produce elemental sulphur have been proposed, though very few have been put into practice. The recovery of sulphur from these process residues is essentially untried and few attractive routes are evident.

The objective of this testwork was to identify attractive sulphur recovery routes from residues produced by existing and proposed hydrometallurgical process. Treating two types of residue can be envisaged: primary residues such as Cominco's zinc pressure leach (ZPL) residue or a ferric chloride leach residue of lead concentrate, and secondary residues such as Inco's nickel sulphide anode slimes.

The three types of process which can produce the primary and secondary residues are described below.

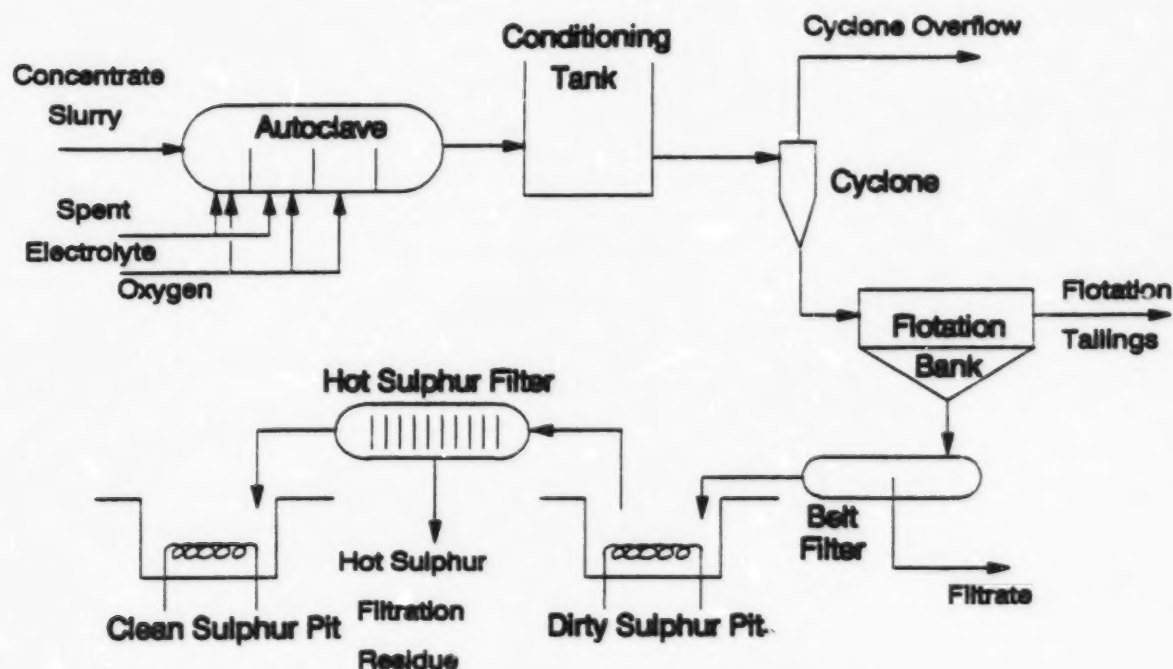
Cominco Zinc Pressure Leach

The zinc pressure leach process consists of a continuously fed autoclave operating at approximately 1.1 MPa (total pressure), 0.65 MPa oxygen (partial pressure) and 140°C to 150°C. Preheated spent zinc electrolyte, zinc concentrate slurry and oxygen are fed to the autoclave, where the temperature is maintained by the exothermic oxidation of sphalerite. The presence of soluble iron species helps transfer electrons and increases the rate of reaction:



Some sulphur is also oxidized to sulphuric acid and some of the soluble iron is precipitated as jarosites of lead, zinc, sodium, potassium, ammonium, hydronium, thallium and silver. The leached slurry is discharged into a flash tank and then fed to a conditioning tank where the sulphur pellets start to convert to the orthorhombic allotrope. From the conditioning tank the slurry is fed to cyclones where jarosite and very fine sulphur report to the overflow, and sulphide-coated sulphur pellets report to the underflow. The sulphur in the underflow is further upgraded by flotation. The filtered concentrate is then fed to a melting pit and filtered at 150°C to obtain the final sulphur product. The simplified flowsheet is shown below.

FIGURE A Cominco Zinc Pressure Leach

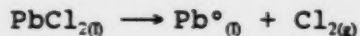


Ferric Chloride Leach of Lead Concentrates

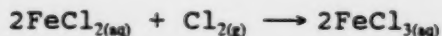
Various processes involving chloride leaching of lead concentrates have been proposed. Essentially, most of them are a ferric chloride leach and would produce similar residues containing elemental sulphur, "unreactive" sulphides (pyrite, chalcopryrite) and gangue. A typical process involves leaching lead sulphide with ferric chloride at about 100°C.



Sufficient chloride is added to the lixiviant to maintain the solubility of the lead chloride complex. Cooling the leach liquor crystallizes lead chloride. The lead salt is filtered, washed and dried and is fed to a fused electrolysis cell to recover metallic lead.

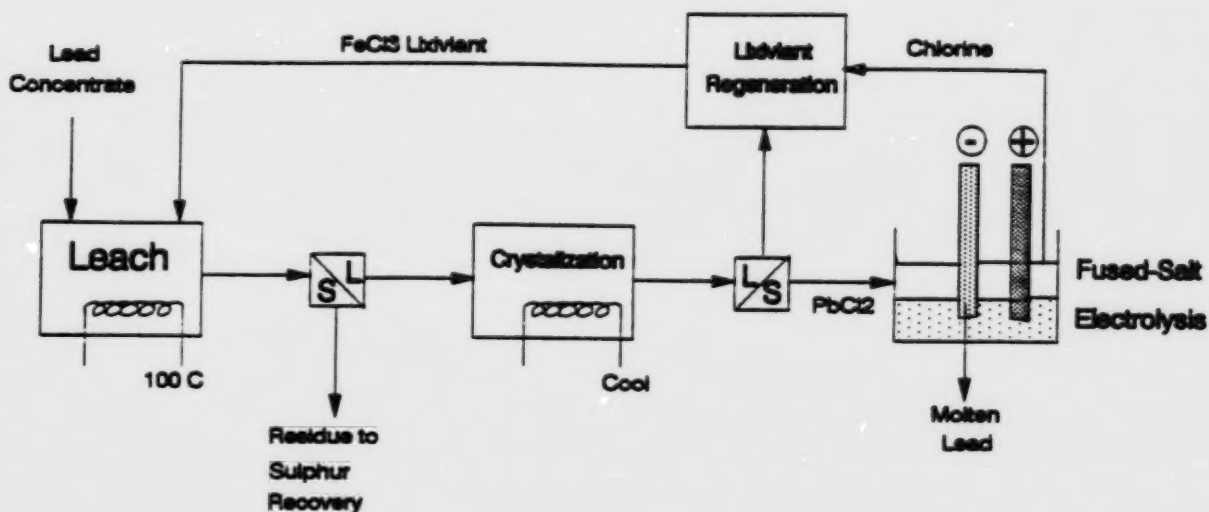


The chlorine is collected for regeneration of the lixiviant.



A simple flowsheet is shown below.

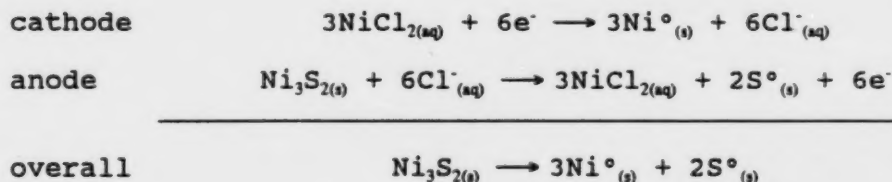
FIGURE B A Ferric Chloride Leach Route for Lead Concentrates



Inco Thompson Nickel Refinery

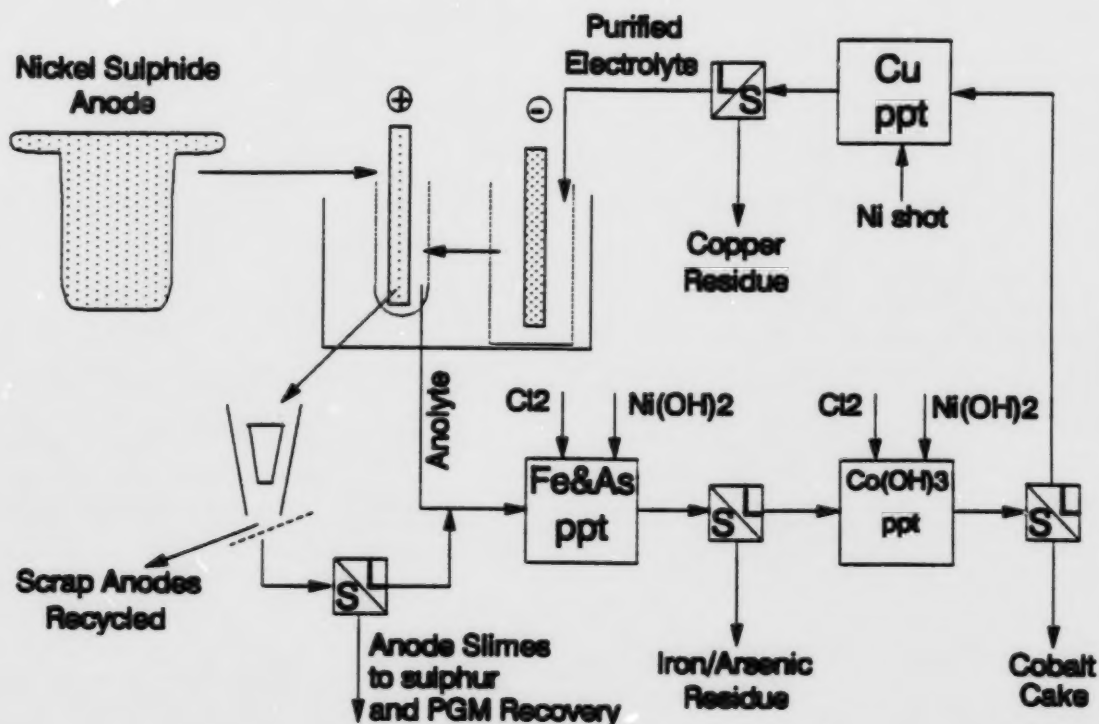
Molten nickel matte from converters is fed to anode molds where the matte is allowed to cool slowly in order to prevent anode cracking. Once cool, the nickel sulphide anodes are electrorefined. The electrolytic cells consist of nickel cathodes (made from starter sheets) in a diaphragm box and the anodes in a loose weave bag to catch falling slimes. Purified chloride electrolyte is fed to the

cathode diaphragm boxes where nickel is electrowon. The resulting catholyte passes through the diaphragm and through the loose weave bags into the anolyte where the nickel in the nickel sulphide anodes is dissolved.



Imbalances in the anode and cathode current efficiencies must be addressed. The resulting anolyte is drawn off, filtered and purified to remove iron, arsenic, cobalt and copper. The spent anodes are crushed, washed and screened to recover the anode slimes. Elemental sulphur can be recovered from the slimes, leaving a residue rich in precious metals.

FIGURE C Nickel Refinery Flowsheet



OUTLINE OF THE INVESTIGATION

Five methods of sulphur separation were investigated: cycloning, pelletization, hot sulphur filtration, solvent leaching and aqueous leaching. Three sulphide residues were investigated: hot sulphur filtration residue from Cominco's zinc pressure leaching process, Inco nickel sulphide anode slimes and a residue resulting from a laboratory ferric chloride leach of lead concentrate.

a) Cycloning of Secondary Sulphur Residues

The effect of grind on the recovery of sulphur by cycloning from both a hot sulphur filtration residue and nickel sulphide anode slimes provided by Inco was examined. The effects of vortex and apex sizes, and solids density, were tested.

b) Pelletization of Secondary Sulphur Residues

The upgrading of the two sulphur residues by pelletization was examined. The effects of adding Lignosol® surfactant and of the autoclave conditions were determined.

c) Hot Filtration of Primary Sulphur Flotation Concentrate

A laboratory hot sulphur filter was developed in conjunction with another sulphur project. The commissioning of the equipment was carried out with a plant sulphur flotation concentrate resulting from Cominco's zinc pressure leach residue.

d) Carbitol Leaching of Secondary Sulphur Residues

The leaching with carbitol of sulphur from the hot sulphur filtration residue and of Inco nickel sulphide anode slimes was examined through several cycles. The effects of temperature and the water content in carbitol on sulphur solubility were determined.

e) Ammonium Sulphite Leaching of Secondary Sulphur Residues

The feasibility of using an aqueous solution of ammonium sulphite as a lixiviant for sulphur was determined. The ammonium sulphite solution was obtained from Cominco's acid plant tail gas scrubber.

f) Recovery of Sulphur from a Residue of a Ferric Chloride Leach of Lead Concentrate

A brief comparison of four lixiviants for sulphur recovery from a ferric chloride leach of lead concentrate was carried out. The four lixiviants were: a lime slurry, carbitol, ammonium sulphide and ammonium sulphite.

SUMMARY OF THE TESTWORK RESULTS

a) Literature Survey

Approximately a thousand references were located in 21 data bases; of which about 200 were relevant and not duplicated. Of the approximately 200 references, 136 described the production of residues by leaching sulphidic materials; the majority involved leaching concentrates in sulphate media under pressure (45 references) or atmospheric chloride leaching (49 references). Very few of the 136 references addressed the problem of the recovery of elemental sulphur from the leach residues. Other lixiviant media that were mentioned included fluosilicic, acetic, and nitric acids, ammonia, acetonitrile, and perchlorate and sulphate solutions. Of the 200 references, only about thirty were directed at or were relevant to the recovery of sulphur.

A list of the references can be found in the bibliography.

b) Cycloning

The effect of the grind on the recovery of elemental sulphur by cycloning from both hot filtration residue and Inco nickel sulphate anode slimes was investigated. At best, only modest upgrading of elemental sulphur at low recoveries could be achieved.

Grinding the hot sulphur filtration residue and cycloning upgraded the sulphur from 44% S° to 70% S°, but with only a 78% recovery to the overflow. The Inco nickel sulphide anode slimes also gave poor sulphur recoveries on cycloning.

c) Pelletization

The objective of pelletization was to melt the elemental sulphur, disperse it into droplets so that on cooling a clean sulphur pellet was obtained; or was clean internally so that the pellet surface could be cleaned by attrition. The hot filtration residue, when pelletized, would yield an internally clean sulphur pellet but with

only a little upgrading in the pellets and with minimal recovery. The nickel sulphide anode slimes (69% elemental sulphur), when pelletized, resulted in a modest upgrading, with 90% of the sulphur reporting to the plus 0.15 mm fraction assaying 86% S°.

d) Hot Sulphur Filtration

In three tests (essentially scouting tests), two sulphur pellet flotation concentrates (80%-90% S°) were filtered hot (ca 140°C). Laboratory results indicated that filtration residues as low as 25% S° could be achieved. Contemporary plant performance leaves at least 40% S° in the hot filtration residue (HFR).

e) Carbitol Leaching

The results of leaching of sulphur from the hot sulphur filter residue (44% elemental sulphur) in 2(2-ethoxy-ethoxy)ethanol (carbitol) and allowing the sulphur to recrystallize were encouraging. Over 70% of the sulphur could be recovered in the plus 74 micrometre fraction as plus 90% elemental sulphur. Further testwork using carbitol and Inco nickel sulphide anode slimes showed that a good sulphur recovery is possible, but a careful water balance will be necessary.

The presence of water was found to decrease significantly the solubility of sulphur in carbitol. Sulphur solubility data in carbitol/water mixtures were generated.

f) Ammonium Sulphite Leaching

Ammonium sulphite is an excellent lixiviant for elemental sulphur, and is available at plants that scrub SO₂ tail gas with ammonia. The bulk of the testwork was performed using hot sulphur filtration residue. Elemental sulphur extractions from 85%-99% were achieved, and the resulting leach liquor may be suitable for fertilizer production. Elemental sulphur (90%-99% pure) might be recovered from the leach liquor after acidification to produce pure SO₂ gas, ammonium sulphate solution and elemental sulphur.

The major disadvantage of ammonium sulphite solution as a lixiviant was the contamination of the leach liquor by an unidentified soluble lead species. Consequently, the leach liquor required purification prior to sulphur precipitation or use as a fertilizer.

g) Comparison of Four Lixiviants

The initial ferric chloride leach tests on Sullivan and Ronaldo Gubbins Trade (RGT) lead concentrates confirmed the high recoveries of lead (>95%) observed in the past. Subsequent tests showed:

- (1) Sulphur was not significantly upgraded during conventional flotation of the leach residue.
- (2) Goulac added during the leach to help the subsequent recovery of sulphur possibly inhibited lead leaching without aiding sulphur recovery.

A sulphur bearing residue from the ferric chloride leach of Sullivan lead sulphide concentrate was leached using four lixiviants.

The four sulphur lixiviants examined were: lime slurry, carbitol, ammonium sulphide solution (200 g/L), and an ammonium sulphite solution (approximately 500 g/L).

- (1) Good sulphur leaching (>95%) was achieved with all four lixiviants.
- (2) On acidification, the filtered leach liquor from the lime slurry leach resulted in an overall sulphur recovery of 60%. The theoretical maximum of 75% is due to some sulphur being volatilized as hydrogen sulphide and sulphur dioxide. The sulphur product assay was better than 99% S°.
- (3) Carbitol leaching at 140°C and crystallizing the sulphur from the filtrate at 10°C resulted in an overall sulphur recovery of 96%.
- (4) Boiling off H₂S and NH₃ from the ammonium sulphide leach liquor resulted in close to a 100% recovery of the sulphur. Product assay was again better than 99% S°.
- (5) Ammonium sulphite leaching also resulted in a high product recovery, but the assay was low (about 95% S°). Sparging some H₂S through the filtrate to precipitate lead sulphide prior to sulphur precipitation improved the sulphur quality.

CONCLUSIONS AND RECOMMENDATIONS

Physical methods such as, grinding/cycloning and pelletization, did not upgrade sulphur from secondary sources. A possible explanation is that sulphur in secondary residues is too intimately dispersed with the gangue material. Physical methods for separating sulphur from Inco nickel sulphide anode slimes or Cominco's hot filtration residue are unlikely to succeed. This need not be the case with all secondary sulphur residues or primary residues. For example, the sulphur pellets coming from Cominco's zinc pressure leach plant are upgraded by cycloning, flotation and elutriation of fines through a filter. Initial commissioning tests of the laboratory hot sulphur filter proved encouraging. Some further tests using Inco nickel sulphide anode slimes are justified.

Carbitol is miscible with water, and is a reasonable solvent for sulphur, consequently, the residues do not have to be completely dry before sulphur lixiviation, and water might be used to wash carbitol from the leached residues. A careful water/carbitol balance will be required, as well as reflux distillation to recover carbitol from waste water streams. The presence of water reduces the solubility of sulphur in carbitol.

Ammonium sulphite is a good lixiviant for sulphur but some considerations are given below:

- (1) Ammonium sulphite leach solution is not recoverable, but it can be converted to ammonium sulphate fertilizer.
- (2) Ammonium thiosulphate solution, an intermediate during sulphur production, is close to fertilizer grade.

Ammonium sulphite lixiviation of sulphur is location specific, i.e., close to fertilizer manufacture and ammonium sulphite production, such as Cominco's Trail operation.

Lime as a lixiviant for sulphur is satisfactory, though purification of the sulphur product may be required. Lime is very process specific; i.e., the process must use a medium which solubilizes calcium - preferably nitrate because nitrogen oxides can be recycled to a primary leach.

Ammonium sulphide was confirmed as an excellent lixiviant for elemental sulphur. The main concern, however, is the extreme toxicity of ammonium sulphide solutions and vapours.

ACKNOWLEDGEMENTS

The experimental testwork was carried out by I.R. Franklin, T.S. Jankovic, D. Comba, M. DeRosa, and P. Checknita. The author wishes to extend his thanks to E.G. Parker for his help and guidance, and to D.R. McKay for his encouragement.

DETAILED DESCRIPTION OF THE TESTWORK

(a) Literature Survey

The computer literature strategy is summarized in Figure 1. The word sulphur (or sulfur) had to be qualified by the term elemental, otherwise a prohibitively large number of references would have been picked up. Approximately a thousand references were located in 21 data bases, of which about 200 were useful and not duplicated. Of the approximately 200 references, 136 references described the production of residues by leaching sulphidic materials; the majority involved leaching concentrates in sulphate media under pressure (45 references) or atmospheric chloride leaching (49 references). Very few of the 136 references addressed the problem of the recovery of elemental sulphur from the leach residues. Other lixiviant media that were mentioned included fluosilicic, acetic, and nitric acids, ammonia, acetonitrile, and perchlorate sulphate solutions. Of the 200 references, about thirty were directed at or were relevant to the recovery of sulphur.

A list of references for the various proposed leach circuits resulting with elemental sulphur in the residue is given in the Bibliography.

In the first survey in November 1987, the keyword 'flotation' was inadvertently omitted. 'Flotation' was included in the second literature survey performed for the period January 1987 to July 1990.

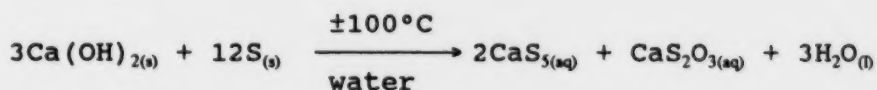
Aqueous Solvents

Ammonium sulphide was proposed as a lixiviant and appears promising. Baker (1) outlined the dissolution of elemental sulphur in aqueous ammonium sulphide and the subsequent recovery of sulphur and lixiviant by thermal decomposition. Kunda (2) described two alternatives to thermal decomposition for sulphur recovery: acidification and oxidation. Haver (3) also outlined sulphur recovery from residues resulting from ferric chloride leaching of chalcopyrite. A similar route was investigated by Ravi (4). The

analogous dissolution of sulphur with sodium sulphide has been described by Lei (5).

Mack (6) describes the production of ammonium thiosulphate fertilizer solution by leaching sulphur in ammonium sulphite. This process may be applicable in certain applications for sulphur recovery in the metallurgical industry. Three patents (7,8,9) describe processes for the recovery of elemental sulphur from thiosulphate solutions.

Elemental sulphur is leached in a lime-water slurry (10), and the equation is given by



Dissolution of sulphur as the polysulphide may have applications under certain circumstances. Weston (11) describes the use of aqueous solutions of calcium sulphide or calcium hydrogen sulphide for the recovery of sulphur. The sulphur could be precipitated simply by boiling, thus regenerating the lixiviant. Seidell and Linke (12) list the solubility of sulphur in various aqueous solutions.

Patents by Maree et al. (13) and Satah et al. (14) describe the bacterial anaerobic reduction of sulphate residues (CaSO_4) to H_2S and the aerobic oxidation of H_2S to S^0 . Possibly these two processes could be combined to convert jarositic residues.

Organic Solvents

The petroleum and natural gas industries extensively use organic solvents in sulphur recovery. As an example, three patents (15,16,17) describe the use of an alkylamine and dialkyl disulphide mixture for the unblocking and recovery of sulphur from sour gas well equipment. The description of the prior art gives examples of the use of the aromatic hydrocarbons benzene, toluene, xylene and naphtha as having previously been used or proposed. Also cited in the prior art description was the use of alkylamines (e.g. ethylamine) in basic aqueous solutions, alkyldisulphides and pyridene.

Gordienko (18) and Semenish (19), respectively, investigated the dissolution and crystallization of elemental sulphur from tetrachloroethene solutions. In a patent, Weirman (20) describes

the recovery of sulphur from sulphur ores by leaching elemental sulphur in condensing tetrachloroethene vapours and recovering it as molten sulphur by boiling off the tetrachloroethene. Kunda (2) evaluated several halogenated hydrocarbons for their suitability as a solvent for sulphur leaching.

Tebbe (21) investigated the dissolution of sulphur in methanol and acetonitrile and the resulting sulphur ring conversions in solution. The sulphur solubility in glycol derivatives, an aniline derivative, quinoline and their mixtures was examined by Sciamanna (22). Herve (23) investigated the suitability of several glycol derivatives as sulphur lixiviants for sulphur recovery from a hydrometallurgical leach residue. Dubow (24) describes a process which leaches elemental sulphur using kerosene. In an earlier process of leaching sulphur using kerosene, as described by Simpson (25), it was claimed that this process would be suitable for recovering sulphur from low grade ores. Baur (26) invented a process where H_2S was absorbed in an organic solvent (a glycol derivative was used in an example), and sulphur was precipitated when contacted with SO_2 . The sulphur settled readily in the presence of NH_4SCN .

Seidell (12) listed the solubility of sulphur in a number of organic solvents.

Dubow (24), Weirman (20) and Simpson (25) also described equipment that may be used for the dissolution and crystallization of sulphur. Scott (27) described apparatus for the crystallization of sulphur from a trichloroethene solvent, whereas Dubow (28) leached and recovered sulphur in a unit operation.

The table below summarizes the properties of other proposed sulphur solvents.

Table A
Sulphur Solubilities and Properties for Several Solvents

Solvent	Saturated Solution	Temp. °C	Toxicity	Combustible or Flammable	US\$/tonne
	g/kg Sulphur Solubility				
Aniline	460	130	3	C	1300
Benzene	360	130	3	F	260
Bromoform	950	71	3	-	1500*
Carbitol	44	130	2	Fl.Pt. 94°C	1100*
CCl_4	20	54	3	-	550
C_2Cl_3H	30	80	3	-	840
C_2Cl_4	260	121	3	-	680
Chloroform	24	40	3	-	770
CS_2	92	100	3	F	460
Hexane	50	130	2	F	200
Kerosene	100	130	3**	C	640
NH_3 (liquid)	250	16	3	-	160
Phenol	160	130	3	C	175
Pyridine	980	110	2	F	5700
Tetrabromomethane	700	71	3	-	1500*
Toluene	110	83	3	F	190

3 most toxic
1 least toxic
costs are qualitative

* estimated price (1988)
** if used sensibly not overly toxic

Pyridine appears to be an ideal sulphur solvent; it has a high S° solubility and moderate toxicity, but it is very expensive and flammable. Most of the other solvents are toxic to some degree. Hexane, kerosene and carbitol are relatively innocuous and inexpensive, and sulphur solubility is adequate. Hexane is the cheapest but is very flammable. Kerosene has a moderate price and probably is worth further investigation. Carbitol has the advantage of being water miscible; therefore, it should be relatively easy to wash from residues. None of the solvents appears ideal for a given application and some compromise will be necessary. Other factors should also be considered; e.g., reagent stability and environmental acceptability. For example, the chlorocarbons if used wisely should not present a hygiene hazard, but they can degrade, causing severe corrosion problems, and generally are environmentally undesirable.

Physical Sulphur Recovery

The recovery of zinc and sulphur by pressure leaching and its integration into the existing zinc plant at Cominco was described by Whiteside (29). Martin (30) described two alternative sulphur recovery processes at Cominco where elemental sulphur could be produced from a pressure leach residue. The pressure leach residue could be discharged into a pressurized decantation vessel where clean liquid elemental sulphur could settle out and be discharged into a storage pit when required. Alternatively, the residue could be flash discharged and sulphur recovered by cycloning, flotation and filtration. In either case, the sulphur is then pressure filtered at elevated temperature to remove entrained gangue. The process by which sulphur was pelletized during the leaching of zinc sulphide concentrates was patented by Landucci (31). A similar method for recovering sulphur from pyrrhotite was described by Nashner (32). A separating vessel for elemental sulphur resulting from base metal leach residues was shown in a patent by Blanco (33). Narain (34) showed that the recovery of sulphur using sulphur volatilization from copper residues, produced in the Cymet process, was almost complete.

A US patent by Rice and May (35) describes the recovery of elemental sulphur from spent catalysts using liquid ammonia.

Flotation

Narain (36) also investigated the flotation of sulphur from a Cymet-type residue and its subsequent dissolution in ammonium sulphide solutions. The recovery and upgrading of sulphur have been carried out on nickel-pyrrhotite concentrate pressure leach residues (37,38) using flotation. Matthew and Mangano (39) described a sulphuric acid leach wherein nitrate ions were used for electron transfer; their leach residue was up-graded by flotation

to obtain a sulphur concentrate which could be subsequently hot filtered. A comprehensive patent (40) describes the use of various collectors and additives for the recovery of sulphur from materials other than sulphidic residues.

(b) Cycloning

Small scale cycloning tests with both the ground hot sulphur filter residue and Inco nickel sulphide anode slimes proved disappointing. In Table 1, Tests 1.1 to 1.4, a lightly ground material 61% minus 44 μ m was not upgraded significantly. The best result showed an upgrading of the residue from 44% S° to 55% S° with a recovery of 52% in the cyclone overflow. On grinding the residue to 94% minus 44 μ m, cycloning improved the sulphur grade from 44% S° to 70% S° with a 78% recovery in the overflow (Test 1.5); however, the mass balance in this particular test was poor.

Inco nickel sulphide anode slimes as-received had the following size distribution:

Table B

Size Distribution of Inco Nickel Sulphide Anode Slimes

<u>Size</u> (mm)	<u>Fraction Retained</u> (%)	<u>S° Assay</u> (g/kg)	<u>S (total) Assay</u> (g/kg)
+1.651	3.5	92	314
-1.651 +0.595	5.0	400	550
-0.595 +0.212	13.3	785	830
-0.212 +0.149	8.0	789	829
-0.149	70.2	<u>755</u>	794
Total		721	

The minus 0.149 mm fraction was cycloned at two pressures and a modest upgrading could be achieved from 75.5% S° to 85.4% S° with a 75% recovery to the overflow. Two tests are reported in Table 2. The nickel sulphide anode slimes were then ground to 85% minus 44 μ m and cycloned. No upgrading was achieved and the mass balances were extremely poor; sulphur having appeared to be lost. The results are not reported.

Cyclone tests were carried out on bench scale glass cyclones. A schematic diagram is shown in Figure 2.

(c) Leaching Pelletization

Slurry samples were introduced into an autoclave and sufficient agitation was provided to suspend the solids. The temperature was brought up to above 125°C for at least 15 minutes to melt the sulphur. If the molten sulphur settled, the agitation was increased. The slurry was first cooled to 120°C. The temperature of the slurry was allowed to decrease slowly (<1°C/min) to 110°C, thereafter the slurry was allowed to cool quickly. The pelletized slurry could then be removed by suction from the autoclave.

The objective of the pelletization tests was to agglomerate the elemental sulphur into relatively clean sulphur pellets. This was accomplished by heating the residue slurry under pressure to above the melting point of sulphur. Whilst agitating the slurry, the molten sulphur coalesced into relatively large droplets. Upon slow cooling of the slurry, sulphur pellets formed. Ideally, these pellets would be clean on the inside and the surface coated with a minimum of gangue material so that the surface could be cleaned by attrition, if necessary. In later tests the conditions were such that the gangue sulphides could be leached prior to pelletization.

Initial scouting tests with hot sulphur filtration residue proved disappointing (Table 3, Tests 3.1 to 3.7). The best result was 15% recovery of the elemental sulphur, upgrading the residue from 44% S° to 64% S° in the plus 0.149 mm fraction. In Test 3.13, the repelletization was carried out in return acid diluted approximately 50% and in the presence of about 15 g/L ferric under oxygen pressure. The plus 0.149 mm and minus 0.149 mm fractions appeared to be upgraded marginally, though only about 4% of the elemental sulphur was recovered in the plus 0.149 mm fraction. Lignosol surfactant additions varying between 0 and 2 g/kg (S°) were used; no correlation between pelletization and Lignosol surfactant addition was noted. The interior of the pellets was clean but the pellets were coated with unreacted sulphides.

In one test, a zinc pressure leach residue after cycloning and flotation was repelletized. The cleaner concentrate was upgraded from 80% S° to 87% S° in the plus 0.149 mm fraction with a recovery approaching 100%. Again, the pellets were clean with an outside sulphide coating (Test 3.10).

Repelletizing Inco nickel sulphide anode slimes resulted in the following observations. High recoveries (>90%) could be obtained but the sulphur upgrading was modest, from 69% S° to 85% at best. When repelletizing after leaching was done in the presence of ferric and sulphuric acid, similar results were obtained. The Inco nickel sulphide anode slimes slurries required extra agitation (compared to the hot sulphur filtration residue) during the repelletization stage because the sulphur pellets tended to settle

and form a pool of molten sulphur at the bottom of the autoclave. Surfactants were detrimental at high additions (8 g Lignosol/kg S° + 8 g quebracho/kg S°); the S° recovery decreased from >90% to 66% without significantly upgrading the product. The optimum surfactant addition appeared to be 1 g - 2 g Lignosol/kg S°. The results are summarized in Tests 3.8 to 3.17, excluding 3.10 and 3.13.

(d) Hot Sulphur Filtration

A small (pressure) 3 L filter was fabricated especially for hot sulphur filtration. Three commissioning tests were carried out using a sulphur flotation concentrate from Cominco's zinc pressure leach plant at Trail, B.C. The plant hot filtration residues (using a Sparkler® filter) typically assay 40%-45% elemental sulphur with a recovery of about 85% to 90%.

An initial test (Test 4.1), using the 3 L filter, gave a poor elemental sulphur recovery of 83% and a relatively low filtration rate of 26 kg (molten residue)/(m²min). As the experimental technique improved, a relatively low sulphur content in the residue could be achieved (about 24% S°), resulting in a high sulphur recovery of 92% and a fast filtration rate, >74 kg/(m²min) (Tests 4.2 and 4.3).

Filtration was carried out in a pressurized stainless steel filter 8.3 cm in diameter. The filter was heated by steam coils wound around the filter and held in place by Thermon® cement. The sulphur filtrate receiver was also heated by a steam coil tacked and cemented onto the vessel. The vessel was insulated where possible. The fibre glass filter cloth was sandwiched and glued between two asbestos fibre composite gaskets. The gasketed cloth assembly was placed between the flanges of the filter and receiver and was bolted into place. The cloth was supported by a stainless steel mesh (see Figure 4). The general procedure to operate the filter was as follows:

1. Material was melted in a beaker overnight using a silicone oil bath controlled at 150°C.
2. Elemental sulphur was added if necessary; i.e., to reduce the viscosity of the concentrate sample, and allowed to melt.
3. Filter was preheated to 150°C. Whilst preheating, the filter was checked for leaks and correct operation of thermocouples.

4. Molten sulphur was added to the filter, which was then closed and allowed to attain the target temperature.
5. After a preset time (typically 30 minutes), the filter was pressurized to between 500 kPa to 600 kPa gauge.
6. A discharge line under the receiver was opened to allow S° to discharge into a stainless steel collector.
7. A short air blow was allowed. (In later tests the filter was reheated and repressurized to see if additional sulphur could be recovered; none was.)
8. The filter was cooled to approximately 60°C and the receiver and the filter cloth was removed. Hot sulphur filtration residue was removed from the filter body, chiselling caked-on material if necessary.
9. Filtering time and cake thickness were noted.

(e) Carbitol Leaching

Three initial tests, 5.1 to 5.3, to recover sulphur from the hot filtration residue using an in situ dissolution/crystallization technique with carbitol as the solvent proved encouraging. The results are summarized in Table 5.1. Sulphur in the hot filtration cake was dissolved in carbitol at 135°C and allowed to cool slowly, with and without agitation. The resultant crystals were screened at 74 microns and both fractions were weighed and assayed for sulphur. The sulphur products assayed between 88% and 95% sulphur and the recoveries ranged between 68% and 77%. The initial results were encouraging, though no further testwork with hot filtration residues was carried out using this route.

Any residue that will be treated will likely contain water and this will depress the solubility of sulphur in carbitol. Therefore, the effect of water on sulphur solubility in carbitol was measured. Figure 5 shows the sulphur solubility in various carbitol/water mixtures as a function of temperature. Even a small addition of water (5% v/v) depresses the sulphur solubility from about 37 g/L to 27 g/L at 120°C. In practice, a careful water balance will be required.

On contact with sulphur, carbitol develops an unpleasant odour; consequently, degradation of the carbitol was a concern. A sample of carbitol was contacted for approximately three hours with fresh elemental sulphur at 110°C and then cooled to 6°C. This was

repeated four times using the resulting cooled carbitol. No decrease in the sulphur solubility of 31.1 ± 0.4 g/kg at 110°C was noted.

Initial tests in dissolving the sulphur present in Inco nickel sulphide anode slimes in carbitol at 140°C followed by cooling and then screening, gave promising results. Although recoveries were poor, the product purity was in excess of 90% (Table 5.2, Tests 5.4 and 5.5). In Tests 5.6 to 5.10, the overall recoveries exceeded 85% and the recovered sulphur purity was better than 99.7%, except in Test 5.10. In this test, cooling occurred during filtration, with sulphur crystallizing prematurely in the residue, resulting in a poor extraction. Some sulphur also crystallized in the Buchner funnel (steam heated) resulting in a poor mass balance.

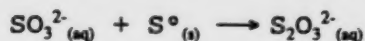
In Tests 5.6 to 5.10, the carbitol was reused and a small amount was added to make up for evaporation losses. The Inco nickel sulphide anode slimes were not dried prior to sulphur lixiviation; consequently, water had to be driven off prior to achieving the target temperature of 140°C during the leach. The water that was collected undoubtedly contained some carbitol; consequently, in practice the carbitol would probably have to be recovered from the evaporate. Careful thought would also have to be given to the recovery of carbitol from the residue and the sulphur product.

The carbitol/water-sulphur solubility tests were carried out in an agitated round-bottom flask under reflux conditions. The dissolution of sulphur was allowed to attain equilibrium for one hour. A sample of the solution was withdrawn with a preheated sawn-off pipette through a filter plug. The sample was then discharged into a beaker (100 mL), weighed quickly and heated in a water bath (40°C - 60°C) until a constant mass was achieved (typically three days). The residue was assumed to be pure sulphur. It was also assumed that no sublimation occurred. Two control tests indicated that there is a slight mass gain due to a carbitol residue, but it would be significant only at the lower sulphur solubilities.

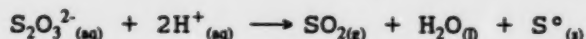
In Tests 5.1 to 5.3, solvent leaching was carried out in a glass autoclave (shown in Figure 5). In Tests 5.4 to 5.10, the leach tests were carried out in a 2 L round-bottom flask with a Teflon® plastic agitator, and heating was supplied by a heating mantle. Water present in the anode slimes was driven off at temperatures up to 140°C and the distillate collected. The hot solvent slurry was filtered in a steam-heated Buchner funnel (13 cm in diameter). Sulphur was allowed to crystallize from the hot filtered solvent at ambient temperature.

(f) Ammonium Sulphite Leaching

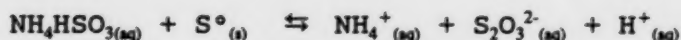
The sulphite leach is based on the reaction



from which sulphur can be recovered by acidification



Initial tests using ammonium bisulphite solution obtained from Cominco's SO₂ tail gas scrubbing plant proved disappointing.

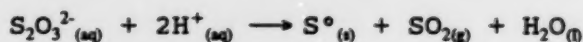


The hydrogen ion liberation inhibited further sulphur dissolution. Tests 6.1 to 6.3, Table 6, summarize the initial testwork. When leaching sulphur in an autoclave with ammonium bisulphite solutions, sulphur dissolution was poor and elemental sulphur was produced during the leach. This was particularly noticeable in Test 6.3. The hot filtration residue gained mass during the leach and the sulphur grade increased from 44% to 48% S⁰, but 30% of the initial sulphur present in the residue could be recovered from the leach liquor. The additional sulphur could come only from the sphalerite in the hot filtration residue or from the SO₃²⁻ in the leach solution. No explanation of this phenomenon is given but it may be worth further investigation.

Pure elemental sulphur (Test 6.4) or hot filtration residue (Test 6.5) were leached in a sodium sulphite solution. These tests confirmed that elemental sulphur could be leached in sulphite solutions. Consequently, two scouting tests were carried out using the tail gas scrub liquor which had been neutralized with ammonium hydroxide to about pH 7. The results are summarized in Tests 6.6 and 6.7.

Several tests to produce solution for sulphur recovery showed that good sulphur leaches could be obtained regularly, Tests 7.1 to 7.4, Table 7. The sulphur recovery from the resulting leach liquors varied from 38% to 122%. The leach liquors were acidified in an autoclave and the sulphur dioxide generated was trapped in a sodium hydroxide solution or hydrogen peroxide. The autoclave was sparged with nitrogen to remove the sulphur dioxide. Once the acidification was complete, the autoclave was pressurized and heated to melt and to pelletize the sulphur. The sulphur produced

heated to melt and to pelletize the sulphur. The sulphur produced this way appeared clean, with impurities reporting in the sulphur pellet at the crystal boundaries. Semi-quantitative spectrographic analysis identified the main impurities as copper and lead. The sulphur dioxide recovered was approximately one quarter of that expected from the equation:



A duplicate set of tests, 7.5 to 7.14, showed that the optimum hot filtration residue concentration in leach solution was about 300 g/L. The maximum leached sulphur concentration was about 145 g/L, and this value agrees well with the estimated SO_3^{2-} concentration of 4.5 mol/L. The elemental sulphur remaining in the residue versus time is given below for three tests:

Table C

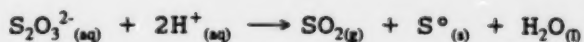
Effect of Time on the Sulphur Content in the Residue
During Sulphite Leaching

Feed Solids (g/L)	Residue S ⁰ Assay (%)		
	200	300	300
Time (min)			
5	0.72	1.25	8.08
15	0.13	0.92	4.99
30	0.27	nd	-
60	-	0.37	0.8
120	0.18	-	-
Grind*	F	G	I

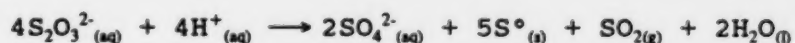
* for grind see Table 13, nd = not detected

The sulphur leach was effectively complete in five minutes when excess ammonium sulphite was used. When close-to-a-stoichiometric amount of sulphite and sulphur were contacted, the rate of leaching seemed slower and excessive time may have allowed the reprecipitation of some sulphur.

In Tests 7.1 to 7.4, it was noticed that substoichiometric amounts of sulphuric acid were required to maximize sulphur recovery for the equation.



Consequently, the effect on sulphur recovery of acid addition to the thiosulphate liquor was investigated. Tests 8.1 and 8.2 showed that the sulphur recovery peaked at 200 mL/L to 300 mL/L addition of 50% v/v H_2SO_4 solution. This is approximately half of the acid required according to the above equation. Another possible equation might be:



This equation would explain the low SO_2 recoveries previously observed.

In Tests 8.1 and 8.2, low sulphur product grades (84%-97% S^0) were obtained, and the major impurities detected by XRD were $(\text{NH}_4)_2\text{Pb}(\text{SO}_4)_2$ and PbSO_4 . Several methods for purifying the ammonium thiosulphate solution prior to sulphur precipitation were investigated. Low temperature purification by heating the thiosulphate leach to 80°C and filtering, then acidifying at an elevated temperature of 125°C under pressure, resulted in high sulphur recoveries but poor final sulphur grades (Tests 8.3a to c). Low temperature purification and acidification at 80°C resulted in poor sulphur grades and low sulphur recoveries (Tests 8.3d to f). In Tests 8.4b to e, the purification was carried out at 125°C under pressure and the sulphur precipitated at 80°C, resulting in a high purity product (>98% S^0) and a good sulphur recovery (101% to 109%). In Test 8.4d, the purification residue was analyzed by XRD. Orthorhombic sulphur, and PbS were identified; XRF showed S, Pb, Sb, Zn and Fe to be present.

In Test 8.4d, the lead concentration decreased from 7.3 g/L to 0.069 g/L in the ammonium thiosulphate solution during purification. About 30 g/L of residue precipitated assaying 69% elemental sulphur.

A quick scouting test showed that the Inco nickel sulphide anode slimes were amenable to sulphite leaching, Test 7.5. Further testwork would be required to establish which other metals also leached.

Table 9 presents a mass balance for the proposed process to treat hot sulphur filtration residue. The flowsheet is shown in Figure 6. The balance is based on actual assays for Test 9. Stream 9.1 is the bisulphite liquor resulting from Cominco's tail gas scrubbing operation. This was neutralized with ammonium hydroxide to pH 9.5, though in practice ammonia gas would be used. The sulphur feed, stream 9.3, was hot sulphur filtration residue, and this was leached with the ammonium sulphite solution at 60°C, close to the natural temperature, for one hour. Only 78% elemental sulphur extraction was obtained, but complete conversion of sulphite to thiosulphate was achieved in stream 9.6. This phenomenon cannot presently be explained, but it is reflected in the apparently high sulphur recoveries. The ammonium thiosulphate leach liquor also had leached about 5 g/L lead. By holding this solution at 100°C for one hour the bulk of the lead could be precipitated as lead sulphide. The purification residue contained 73.6% lead, 6.1% zinc, and 19.3% total sulphur of which 4.0% was elemental sulphur. The purified solution still contained about 0.5 g/L zinc at pH 8.8. On acidifying the purified liquor with a 50% substoichiometric quantity of sulphuric acid, 121% recovery of sulphur was achieved based on sulphur leached from the hot filtration residue, though only a 92% recovery was attained, based on the purified solution thiosulphate analyses. Thiosulphate could not be detected at the end of the precipitation, and only about 1.5 mol of SO₂ gas could be collected. The sulphur mass balance around the acidification circuit is poor; approximately 80% of the sulphur can be accounted for. The final acidity of the acidification liquor was pH 2.0. The acidification was carried out at 75°C, and the sulphur was pelletized by heating to 125°C and pelletizing at 110°C.

Initial scouting tests were carried out in a glass autoclave. Later tests were carried out under atmospheric conditions in normal laboratory glassware or polypropylene vessels.

Sulphur precipitations from thiosulphate solutions were carried out in a glass autoclave. The SO₂ evolved was collected in caustic solution or hydrogen peroxide solution and was titrated. The autoclave was sparged with nitrogen to aid recovery of the sulphur dioxide. Pelletization of the recovered sulphur, if required, was carried out using the method previously described.

(g) Recovery of Sulphur From a Lead Leach Residue

In order to study the recovery of sulphur from a primary leach residue, lead concentrates were leached with hot ferric chloride solution. Lignosol surfactant was added to see if it would aid leaching and liberate sulphur for subsequent flotation.

The leach procedure was as follows:

- (1) The lixiviant pH 0.3 (30 L), containing 25 g/L Fe^{3+} , 254 g/L NaCl was heated to 95°C.
- (2) The required amount of lead concentrate (1.77 g Pb/1 g Fe^{3+}), was added.
- (3) Lignosol surfactant was added if required.
- (4) The temperature was maintained at 95°C with titanium steam coils whilst agitating the leach slurry hot for about 30 minutes.
- (5) The slurry was filtered hot through a preheated pressure filter.
- (6) The residue was then displacement washed using 3 L of hot brine solution (95°C) and 3 L of hot water (95°C).
- (7) The residue was then repulped in 1 L of hot water, filtered and displacement washed with 1 L of hot water again.
- (8) The residue was sampled for moisture content and assayed.

After the residue's moisture content had been determined a "standard" flotation was carried out using the following conditions:

- (1) 200 g (dry mass) of the moist residue was repulped in a 1.2 L stainless steel flotation cell at 1000 rpm.
- (2) The temperature of the slurry was adjusted to 35°C, and at time zero, air was turned on.
- (3) For 1.5 minutes a rougher concentrate was obtained.
- (4) The air was turned off, the slurry was bulked to the original level, and 1 mL of 0.004 g/L Dowfroth 250® frother was added.

- (5) The slurry was conditioned for 2 minutes.
- (6) The roughing continued until more frother was required or the froth was clean.
- (7) If more frother was required, then the conditioning step was repeated.
- (8) Typically, the total roughing time was 6 minutes.

The full results of the leaching of Sullivan lead concentrate and the flotation of the subsequent leach residue are given in Tables 10.1 to 10.4. A summary of some of the more important parameters are given below.

Table D
Summary of Lead Sulphide Leach and Residue Flotation Results

Table	Lignosol Added (g/L)	Sulphur Conversion to S ⁰ (%)	Sulphur Grade (g/kg)			Metal Leached		Department to Float Tails	
			Flotation Conc S _(net)	Flotation Conc S ⁰	Flotation Tails S _(net)	Pb %	Ag %	Pb %	Ag %
10.1	0	52	567	324	58	99	83	0.03	1.6
10.2	0.2	50	491	260	81	92	72	0.05	2
10.3	0.1	52	502	268	140	92	72	0.8	2.6
10.4	0	55	511	282	17	95	75	0.03	0.5
11.1	0	42	519	250	177	97	67	1.2	7.3

The leaching was carried out on a relatively large scale; consequently, it is impossible to duplicate the leaching conditions closely. Taking the above results at face value the following observations can be made:

- (1) The leaching of lead and silver was slightly inhibited by the presence of Lignosol surfactant.
- (2) The presence of Lignosol surfactant during leaching marginally depressed the conversion of sulphide to elemental sulphur and the subsequent recovery of total and elemental sulphur to the flotation concentrate.

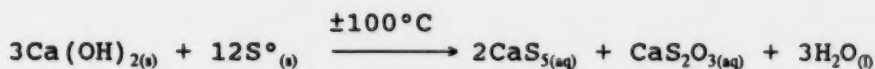
- (3) The deportment of total sulphur, lead and silver to the flotation tailings increased when Lignosol surfactant was present during leaching.
- (4) The sulphur residue was upgraded by a few percent to only about 50% total sulphur. The concentrate was unsuitable for hot filtration.

For comparison with the test with the Sullivan lead concentrate, a test with Ronaldo Gubbins Trading lead concentrate was carried out. The results are given in Table 11.1. Results similar to those realized with the Sullivan lead concentrate testwork were obtained.

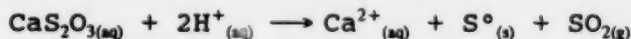
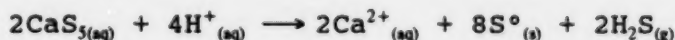
Sulphur Lixiviation

Four sulphur lixiviants were examined for the recovery of sulphur from the leach residue. The lixiviants considered were an aqueous slurry of lime, carbitol, an aqueous solution of ammonium sulphide and an aqueous solution of ammonium sulphite.

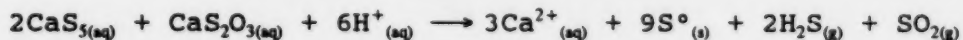
Lime Leach: The recovery of sulphur from metallurgical residues might be considered with nitrate-based leach media. Sulphur-containing residues are leached as the polysulphide,



A major portion of the solubilized sulphur can be recovered as elemental sulphur by acidification.



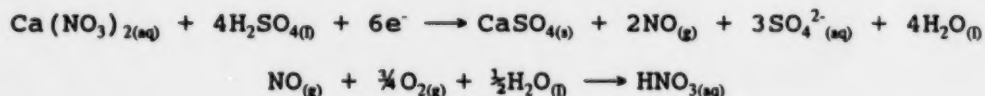
overall



Taken to this stage the maximum sulphur recovery is 75%. Further sulphur recovery should be feasible via a wet Claus reaction,



The acid used to recover sulphur must solubilize calcium, otherwise the calcium salt will contaminate the sulphur product, i.e. sulphuric acid cannot be used at this stage. Nitric acid, if used, could possibly be regenerated via a typical nitric acid leach route,



Procedure for the lime leach was as follows:

- (1) 100 g of residue from Test 10.4 was slurried with 500 mL of water in a beaker.
- (2) 25.7 g of calcium hydroxide was added (1 g $\text{Ca}(\text{OH})_2$ /1 g S°).
- (3) The slurry was agitated for 1 h at 100°C, using a magnetically stirred hot plate.
- (4) The slurry was filtered and washed.
- (5) The filtrate was acidified with hydrochloric acid to pH 2 at 90°C.
- (6) The resulting sulphur was filtered off and washed.
- (7) The filtrate was further acidified to pH 0.5 at 80°C, and the precipitated sulphur was collected and combined.

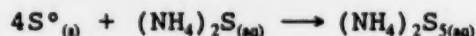
The dissolution of sulphur was good, 97.5%, but the subsequent recovery from solution was poor, 61%, due to the volatilization of hydrogen sulphide and sulphur dioxide. The theoretical maximum recovery from solution was 75%. The overall sulphur recovery for Test 12.1, Table 12, was 59%.

Carbitol Leach: Sulphur was recovered by dissolving elemental sulphur in hot carbitol, filtering hot, cooling, allowing the sulphur to crystallize out, and filtering off the sulphur; the details are given below:

- (1) 100 g of residue was slurried in 800 mL of carbitol in a beaker.
- (2) The slurry was heated to 140°C and agitated using a magnetically stirred hot plate for 15 minutes.
- (3) The slurry was filtered using a steam heated Buchner funnel. The residue was then washed with water and dried.
- (4) The carbitol filtrate was cooled to 10°C so that the sulphur could crystallize.
- (5) The sulphur was filtered and washed with water and then dried.

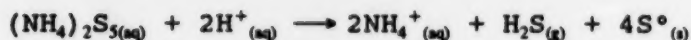
The dissolution of sulphur and the recovery from the carbitol were relatively poor, 92% in both cases (Test 12.2). The apparently poor leaching was ascribed to poor filtration and the slurry cooling prematurely. The poor recovery from the carbitol was probably due to the solubility of sulphur in carbitol at 10°C. The test was repeated using the filtrate from Test 12.2 and bulking up to 800 mL with fresh carbitol. The repeat test gave improved recoveries, 98.9% in leaching and 96% in crystallizing; i.e., an overall recovery of 95% (Test 12.3).

Ammonium Sulphide Leach: The recovery of sulphur by leaching with ammonium sulphide solution has been proposed. The sulphur is leached as a polysulphide in a manner analogous to lime leach:

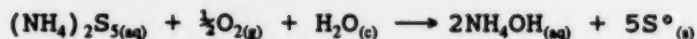


The sulphur can be recovered in three ways,

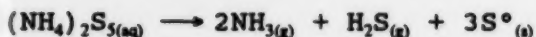
acidification:



oxidation:



boiling:



The exact stoichiometry of the polysulphide formed has not been determined. For the purposes of the scouting tests, an excess of ammonium sulphide was used. Boiling was used to recover the sulphur because the lixiviant can be recovered. In practice, where NH_3 or H_2S might be more readily available or cheaper than power, acidification or oxidation could be considered.

In the two leaches carried out an excess of ammonium sulphide was used to ensure complete dissolution of the elemental sulphur. In both tests, using 360 g/L $(\text{NH}_4)_2\text{S}$ and 240 g/L $(\text{NH}_4)_2\text{S}$, dissolution of elemental sulphur was good (~98%). The recovery of sulphur from solution on boiling was poor (~90%) when the higher ammonium sulphide concentration was used (Test 12.4). This is possibly due to the difficulty in driving off the lixiviant. The sulphur recovery was essentially complete when the lower lixiviant concentration (240 g/L $(\text{NH}_4)_2\text{S}$) was used (Test 12.5).

Ammonium sulphide solution was prepared by sparging 1.0 L of 30% ammonia solution with hydrogen sulphide gas. Once copious quantities of hydrogen sulphide escaped the solution it was assumed that the reaction was complete. The resulting solution assayed 168 g/L sulphur, approximately 5 mol/L, whereas theoretically the value should be nearer 8 mol/L. Either the reaction was incomplete or ammonia had been driven off during the H_2S absorption. The possibility of ammonium hydrogen sulphide formation has to be recognized.

The leach was carried out using the following procedure:

- (1) 100 g of residue was slurried in 300 mL of ammonium sulphide solution. (In Test 12.5, the solution was diluted by 33%.)
- (2) The slurry was agitated and heated at 30°C for two hours using a magnetically stirred hot plate.
- (3) The slurry was filtered and the residue washed; the first 30 mL of wash water was combined with the filtrate.
- (4) The filtrate was then boiled on a magnetically stirred hot plate until all the H_2S had been driven off, typically 60 minutes.
- (5) The precipitated sulphur was washed, dried and assayed.

Ammonium Sulphite Leach: The chemistry of the sulphite leach has been described previously. An initial leach showed that good sulphur recovery (>95%) is achievable when leaching the residue in ammonium sulphite solution (Test 12.6); the sulphite solution was obtained by sparging ammonia into Cominco's tail gas scrubber liquor. The product sulphur quality was relatively poor, 95% S°. The leach was repeated but the resulting solution was purified by sparging with H₂S until further impurity precipitation did not occur. About 7 g/L of impurities precipitated. The purified solution was then acidified for further sulphur recovery. With purification, the product sulphur grade improved to 99.5% S° (Test 12.7).

Sulphur Quality

The sulphur products from the four sulphur leaches:

- Test 12.1 - lime leach
- Test 12.3 - recycled carbitol leach
- Test 12.5 - ammonium sulphide leach
- Test 12.6 - ammonium sulphite leach without purification

were analyzed semiquantitatively using a spectrographic technique for 22 elements. Surprisingly, the sulphite leach, despite containing only 95% elemental sulphur, was relatively low in impurities. The lime leach sulphur product was particularly impure with respect to Ca, Fe and Zn. The results are summarized in Table 12b.

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Table 1

Cyclone/Hot Sulphur Filtration Residue Tests

Test	<u>Size</u>		<u>Product Mass</u>		<u>Product S°</u>		<u>Sulphur</u>		Comment
	<u>(mm)</u>		<u>(kg)</u>		<u>Assay</u>		<u>Distribution</u>		
	Vortex	Apex	U/F	O/F	U/F	O/F	U/F	O/F	
1.1	7.94	2.58	0.132	0.179	359	496	35	65	Cyclone feed 25% solids 50 kPa 61% -44 microns
1.2	7.94	1.70	0.073	0.216	364	462	21	79	
1.3	5.95	2.58	0.180	0.127	364	553	49	52	
1.4	5.95	1.70	0.086	0.178	353	480	26	74	Cyclone feed 15% solids 94% -44 microns
1.5	5.95	2.58	0.041	0.039	356	704	41	78	
1.6	7.94	2.58	0.034	0.01	328	708	58	37	
1.7	7.94	2.06	0.025	0.011	318	693	50	48	

HFR - Hot Sulphur Filtration Residue
 - Grind B, Tests 1.1 to 1.4
 - Grind E, Tests 1.5 to 1.7
 - see Table 13 for grinds
 - ambient temperature
 - 440 g (S°)/kg

Table 2**Cyclone/Inco Nickel Sulphide Anode Slimes**

Test	Size		Product Mass		Product S°		Sulphur		Comment
	Vortex	Apex	Assay		Distribution				
			U/F	O/F	U/F	O/F			
	(mm)		(kg)		(g/kg)		(%)		
2.1	7.94	2.58	0.118	0.113	684	881	46	57	70% -149 microns 30 kPa
2.2	7.94	2.58	0.072	0.142	620	854	28	75	3.5 kPa

- Inco Nickel Sulphide Anode Slimes 690 g (S°)/kg
- The minus - 149 micron fraction of the as received material
- Ambient Temperature

Grind Properties of Inco Nickel Sulphide Anode Slimes

<u>mesh size</u> (μ m)	<u>Grind Time</u> (min)	
	10	20
+104	0.33	0.18
+74 -104	2.3	2.7
+53 -74	6.6	4.3
+43 -53	9.1	7.3
+38 -43	4.9	2.7
-38	76.7	82.7

- Mill - 25 cm ϕ x 35 cm rubber lined rod mill (RLRM #4)
- $\pm$ 30 kg steel rods
 - $\pm$ 2 kg feed
 - $\pm$ 1 L water

Table 3
Summary of Sulphur Pelletization and Leach Tests

Test	Material	Solids	Mass Products		Product S ^o Assay		Sulphur Distribution		Lignosol	Comments
		Initial (g/L)	-0.149 mm (g/L)	+0.149 mm (g/L)	-0.149 mm (g/kg)	+0.149 mm (g/kg)	-0.149 mm (%)	+0.149 mm (%)	Addition (g/kg S ^o)	
										Straight Repelletization Test
3.1	HFR	333	320	13.3		510		5	0	270 kPa O ₂
3.2	HFR	333	294	35.0		643		15	1	270 kPa O ₂
3.3	HFR	333	300	29.3		478		10	0	270 kPa O ₂
3.4	HFR	333	302	27.0		589		11	1	270 kPa O ₂
3.5	HFR	333	310	23.3		479		8	0.5	270 kPa O ₂
3.6	HFR	333	306	27.3		545		10	2	270 kPa O ₂
3.7	HFR	333	307	23.0		490		8	1	270 kPa O ₂
3.8	IAS	333	27.3	277	306	870	4	105	1	270 kPa O ₂
3.9	IAS	333	20.0	293	149	743	1	94	0	304 kPa O ₂
3.10	ZPLCC	333	19.3	307	162	870	1	100	1	304 kPa O ₂ , ZPLCC 830 g/kg S ^o
3.11	IAS	333	34.0	272	395	810	5	96	1	repeat of Test 13.8 with improved agitation
3.12	IAS	333	56.0	240	354	859	9	89	6	304 kPa O ₂
										Combined Leach
3.13	HFR	300	292	10.0	455	507	101	4	2	10 g/L Fe ³⁺ , pH 0.5, 785 kPa O ₂
3.14	IAS	300	33.0	234	291	930	5	104	2	10 g/L Fe ³⁺ , pH 0.5, 785 kPa O ₂
3.16	IAS	300	64.8	196	54.0	893	17	84	8	10 g/L Fe ³⁺ , pH 0.5, 925 kPa O ₂
3.17	IAS	300	104	153	680	900	34	66		8+8 g/kg Quebracho, repeat of Test 3.16

Conditions: 3-L Glass Autoclave
 agitation - off bottom suspension
 +125°C Sulphur Melting Temperature
 +15 min Sulphur Melting Time
 <1°C/min Cooling Rate During Pelletization, 120 to 110°C
 Grind A HFR Material

HFR - Hot Sulphur Filtration Residue 440 g/kg S^o
 IAS - Inco Ni₃S₂ Anode Slimes 690 g/kg S^o
 ZPLCC - Zinc Pressure Leach Cleaner Concentrate
 nb - O₂ Pressure Includes Water Vapour Pressure
 - Combined Leaches - 150°C, 1.5 h

Table 4

Hot Sulphur Filtration Results

Test #2		<u>Mass</u> <u>(g)</u>		<u>Assay</u> <u>Assay S*</u>	<u>Deportment</u> <u>(%)</u>	<u>Cake</u> <u>Thickness</u> <u>(mm)</u>	<u>Cake</u> <u>Density</u> <u>(kg/m³)</u>	<u>Filtration</u> <u>Rate</u> <u>kg/(m²min)</u>
		<u>Total</u>	<u>S*</u>	<u>(g/kg)</u>				
4.1	S* pellets	990	859	868	74	18	2760	26
	S* diluent	300	300	1000	26			
	loss	60	53	892	5			
	S* product	972	967	995	83			
	HFR	250	145	580	13			
					100			
4.2	S* pellets	1000	828	828	100	18	2350	87
	loss	15	12	828	2			
	S* product	761	759	998	83			
	HFR	213	53	248	6			
					100			
4.3	S* pellets	1000	868	868	100	15	2360	74.5
	loss	17	15	868	2			
	S* product	804	799	994	92			
	HFR	178	42	235	5			
					100			

* Ass:med

Tests 4.1, 4.3, July 1989 Zinc Pressure Leach Flotation Concentrate

Test 4.2, October 1989 Zinc Pressure Leach Flotation Concentrate

HFR - Hot Sulphur Filtration Residue

Pressure (gauge) 400 kPa

Table 5.1

Solvent Leach/Crystallization of Hot Sulphur Filtration Residue

Test	Initial (g/L)	Product Mass		Product S ^o Assay		Sulphur Distribution		Comment
		-0.074 mm (kg/L)	+0.074 mm (kg/L)	-0.074 mm (g/kg)	+0.074 mm (g/kg)	-0.074 mm (%)	+0.074 mm (%)	
5.1	100	0.061	0.034	156	882	22	68	No agitation during cooling
5.2	100	0.062	0.035	156	920	23	77	Agitation during cooling
5.3	100	0.063	0.032	184	947	27	73	No agitation during cooling

3-L - Autoclave
 1-L - Carbitol
 135°C - Solubilization Temperature
 15° min - Solubilization Time
 Grind A - Tests 5.1 and 5.2
 Grind C - Test 5.3
 430 g/kg - HFR Head Assay
 - see Table 13 for grinds

Table 5.2**Summary of Sulphur-Carbitol Leaches of Inco Nickel Sulphide Anode Slimes**

Test	Mass (g/L)		Assay (g/kg)		Sulphur Leached		Sulphur Recovered From Solvent			Overall (%)	Comment
	Initial Feed	Final Res	Initial Feed	Final Res	(g/L)	(%)	Mass (g/L)	Assay (g/kg)	Recovery (%)		
5.4	57.5	18.5	770	565	33.8	76.3	33.5	916	90.7	69	Screened 104 μ m
5.5	57.5	30	770	660	15	33.9	22.5	966	145	49	Screened 208 μ m
5.6	57.5	10.5	770	43	43.9	98.98	40.0	998	90.9	90	Filtered
5.7	57.5	15.5	770	178	41.5	93.8	39.5	997	94.9	89	Filtered
5.8	57.5	13.84	770	131	42.5	95.9	38.3	999	90.1	86	Filtered
5.9	57.5	15.5	770	238	40.6	91.7	41.0	997	92.4	85	Filtered
5.10	57.5	21	770	457	34.7	78.3	27.0	999	61	48	S ^o Xtals froze inside Buchner funnel and were lost

Conditions

140°C Solubilization Temperature
 30°C Final Crystallization Temperature
 2-L Solvent Carbitol
 1-h Leaching Time at 140°C

Vessel

3-L Round bottomed flask with heating mantel
 with condensor for water

Table 6

Initial Sulphite-Sulphur Leach Tests

Test	HFR Mass/(g/L)		HFR Assay (g/kg)		Sulphur (S°) Leached		Impure Sulphur Recovery			Comment
	Initial	Final	Initial	Final	(g/L)	(%)	Mass (g/L)	Assay (g/kg)	Recovery (%)	
6.1	221	188	440	360	20	30	50	950	240	60°C 5.5 mol/L NH_4HSO_3
6.2	221	199	440	390	13	21	37	950	270	40°C
6.3	221	229	440	480	-12	-13	41	900	00	80°C
6.4	45°	6	1000	10007	39	78				S°, 1.5 mol/L Na_2SO_3 , 60°C
6.5	100	54	430	19	42	98	35			1.5 mol/L Na_2SO_3 , 60°C
6.6	79	41	430	2	34	99	49	960	144	40°C
6.7	156	85	430	2	67	99	86	970	128	40°

HFR - Hot Sulphur Filtration Residue

Conditions

- 2 h Time - Tests 6.1 to 6.3 Grind A
- 2.4 h Time - Test 6.4 S° Elemental Sulphur
- 1.5 h Time - Test 6.5 Grind C
- 2.5 h Time - Test 6.6 and 6.7 Grind C
- see Table 13 for grind

Table 7

Hot Sulphur Filtration Residue Sulphur Ammonium Sulphite Leach Tests

Test	Mass (g/L)		HFR Assay (g/kg)		Sulphur Leached		Sulphur Recovery			Comment
	Initial	Final	Initial	Final	(g/L)	(%)	Mass (g/L)	Assay (g/kg)	Recovery (%)	
7.1	200	109	440	2.7	87.7	99.7	80	985	89.8	200 mL', SQS Cu Pb main contaminant
7.2	200	106	440	2.7	87.7	99.7	34	980	38.0	500 mL', lignosol added to aid pelletization
7.3	200	106	440	5.1	87.5	99.4	109	975	121.6	300 mL'
7.4	200	105	440	5.9	87.4	99.3	105	985	118.2	300 mL'
7.5	200	104	440	0.8	87.9	99.9				45 min
7.6	250	132	440	31	105.9	96.3				45 min
7.7	300	162	440	36	126.1	95.5				45 min
7.8	350	194	440	75	139.5	90.6				45 min
7.9	400	246	440	128	145.4	82.6				45 min
7.10	200	108	440	.6	87.9	99.9				45 min
7.11	250	142	440	13.5	108.1	98.3				45 min
7.12	300	164	440	12.5	130.0	98.5				45 min
7.13	350	196	440	75	139.4	90.5				45 min
7.14	400	244	440	128	144.8	82.3				45 min

Conditions

HFR - Hot Sulphur Filtration Residue
SQS - Semi-Quantative Spectroscopic Analysis

60°C Temp (except Test 7.2 ambient)
4.5 mol/L Ammonium Hydrogen Sulphite Solution
neutralized with ammonium hydroxide

Grind C Test 7.1
Grind D Tests 7.2 to 7.4
Grind G Tests 7.5 to 7.9
Grind H Tests 7.10 to 7.14
see Table 13 for grind

* Volume of 50% v/v H₂SO₄ added to one litre of leach liquor to recover sulphur.

Table 8

Sulphur Leaching Purification and Precipitation

Test	HFR Mass (g/L)		HFR Assay (g/kg)		Sulphur Leached		Sulphur Recovery			Comment
	Initial	Final	Initial	Final	(g/L)	(%)	Mass (g/L)	Assay (g/kg)	Recovery (%)	
8.1a	300	160*	455	8	135	99	121	940	94	450 mL/L acid addition (50% v/v H ₂ SO ₄)
b					135	99	141	930	97	300
c					135	99	147	970	105	250
8.2a	300	168	445	96	120	88	121	910	92	100 mL/L acid addition (50 v/v H ₂ SO ₄)
b					120	88	165	920	126	200
c					120	88	156	920	120	300
d					120	88	150	900	112	400
e					120	88	130	840	91	500
8.3a	300	186	448	198	114	85	149	900	117	low temp impurity ppt (80°C)
b					114	85	137	860	103	high temp sulphur ppt (125°C)
c					114	85	156	880	121	@ 250 mL/L 50% v/v acid
d					114	85	92	840	67	low temp impurity ppt (80°C)
e					114	85	101	890	78	low temp sulphur ppt (80°C)
f					114	85	108	890	84	@ 250 mL/L 50% v/v acid
8.4a	300	168	455	96	120	88	167	930	129	low temp ppt S° no purification 1 h, 200 mL/L 50% v/v acid
b					120	88	131	980	107	high temp purification 125°C s/L sep, low temp S° 80°C 10/60
c					120	88	131	990	108	high temp purification 125°C s/L sep, 80°C sulphur ppt 20/90 min
d					120	88	122	990	101	repeat 20/45 min
e					120	88	132	990	109	repeat 1.25/1 h
8.5	200	61	711	253	127	89				Inco Ni ₃ S ₂ Anode Slimes

* Estimated

HFR - Hot Sulphur Filtration Residue

ConditionsGrind Test

I 8.1
 - 8.2 5 minute grind, unspecified size
 E&J 8.3 2:1 ratio of E and J grinds
 - 8.4 5 minute grind, unspecified size
 see Table 13 for grind

Test 10.1

Ferric Chloride Leach of Sullivan Lead Concentrate

	Element						
	Total	Pb	Zn	Fe	S ¹⁰⁰	S ⁰	Ag
<u>Feed</u>							
Assay (%)	-	61	7.6	8.6	18.6	-	0.042
Mass (kg)	2.14	1.31	0.163	0.184	0.4	-	0.9 g
<u>Residue</u>							
Assay (%)	-	2.0	18.3	19.2	49.5	31.3	0.0224
Mass (kg)	0.70	0.014	0.13	0.13	0.35	0.219	0.16 g
Department (%)	-	1	80	71	88	55	17
<u>Flot Tails</u>							
Assay (%)	-	0.52	0.48	19.7	5.8	-	0.0226
Mass (kg)	0.065	0.34 g	0.31 g	0.013	3.8 g	-	0.015 g
Department (%)	-	0.026	0.2	7	1	-	1.6
<u>Flot Conc</u>							
Assay (%)	-	2.2	19.1	19.9	56.7	32.4	-
Mass (kg)	0.635	0.14	0.12	0.026	0.36	0.21	
Department (%)	-	11	74	68	90	52	

Conditions:

Leach

Time : 15 min @ 95°C + heating up time
Temperature : 95°C
Initial Solids Content : 78.3 g/L
Agitation : 420 rpm
Agitator : 13.2 cm ø, 6 bladed radial turbine
Solution Volume : 30 L
Lignosol : 0 g/L

Standard Flotation

Solids : 200 g
Volume : 1 L
Agitation : 1000 rpm
Roughing Time : -
Total Time : 6 min

Test 10.2

Ferric Chloride Leach of Sullivan Lead Concentrate

	Element						
	Total	Pb	Zn	Fe	S ¹⁰⁴	S ⁹	Ag
<u>Feed</u>							
Assay (%)	-	61	7.6	8.6	18.6	-	0.042
Mass (kg)	2.14	1.31	0.163	0.184	0.4	-	0.9 g
<u>Residue</u>							
Assay (%)	-	13.0	16.3	18.0	45.1	-	0.0293
Mass (kg)	0.845	0.11	0.14	0.152	0.38	-	0.25 g
Deportment (%)	-	8	84	83	95	-	28
<u>Flot Tails</u>							
Assay (%)	-	0.78	2.4	22.6	8.1	-	0.0267
Mass (kg)	0.076	0.59 g	1.8 g	0.017	6.2 g	-	0.02 g
Deportment (%)	-	0.05	1	9	2	-	2
<u>Flot Conc</u>							
Assay (%)	-	14.4	16.7	17.2	49.1	26.0	0.0305
Mass (kg)	0.767	0.11	0.13	0.13	0.38	0.2	0.23 g
Deportment (%)	-	8	79	72	94	50	26

Conditions:

Leach

Time : 15 min, no heating up time
Temperature : 95°C
Initial Solids Content : 71.3 g/L
Agitation : 420 rpm
Agitator : 13.2 cm ø, 6 bladed radial turbine
Solution Volume : 30 L
Lignosol : 0.2 g/L

Standard Flotation

Solids : 200 g
Volume : 1 L
Agitation : 1000 rpm
Roughing Time : -
Total Time : 6 min

Test 10.3

Ferric Chloride Leach of Sullivan Lead Concentrate

	Element						
	Total	Pb	Zn	Fe	S ^{tot}	S ^o	Ag
<u>Feed</u>							
Assay (%)	-	61	7.6	8.6	18.6	-	0.042
Mass (kg)	2.176	1.33	0.17	0.19	0.404	-	0.91 g
<u>Residue</u>							
Assay (%)	-	11.8	15.9	18.0	44.8	-	0.0293
Mass (kg)	0.862	0.10	0.14	0.16	0.39	-	0.25 g
Deportment (%)	-	8	81	82	96	-	28
<u>Flot Tails</u>							
Assay (%)	-	14.0	29.0	22.6	8.8	-	0.0325
Mass (kg)	0.073	0.01	1.02	0.016	6.4 g	-	0.02 g
Deportment (%)	-	0.8	12	9	1.6	-	2.6
<u>Flot Conc</u>							
Assay (%)	-	13.1	17.3	17.5	50.2	26.8	0.0310
Mass (kg)	0.789	0.10	0.14	0.17	0.4	0.21	0.24 g
Deportment (%)	-	8	80	73	98	52	27

Conditions:

Leach

Time : 15 min, no heating up time
Temperature : 95°C
Initial Solids Content : 71.3 g/L
Agitation : 420 rpm
Agitator : 13.2 cm ø, 6 bladed radial turbine
Solution Volume : 30 L
Lignosol : 0.1 g/L

Standard Flotation

Solids : 200 g
Volume : 1 L
Agitation : 1000 rpm
Roughing Time : 6 min
Total Time : 10 min

Test 10.4

Ferric Chloride Leach of Sullivan Lead Concentrate

	Element						
	Total	Pb	Zn	Fe	S ^{tot}	S ⁺	Ag
<u>Feed</u>							
Assay (%)	-	61	7.6	8.6	18.6	-	0.042
Mass (kg)	2.177	1.33	0.17	0.19	0.405	-	0.91 g
<u>Residue</u>							
Assay (%)	-	8.5	16.1	19.5	46.5	27.1	0.0272
Mass (kg)	0.825	0.07	0.13	0.16	0.38	0.22	0.22 g
Department (%)	-	5	78	85	95	55	25
<u>Flot Tails</u>							
Assay (%)	-	0.63	0.34	24.1	1.7	0.6	0.0063
Mass (kg)	0.074	0.5 g	1.25 g	0.018	1.3 g	0.44 g	0.005 g
Department (%)	-	0.03	0.15	9	0.3	0.1	0.5
<u>Flot Conc</u>							
Assay (%)	-	9.3	17.5	19.1	51.1	28.2	0.0301
Mass (kg)	0.742	0.07	0.13	0.14	0.38	0.21	0.22 g
Department (%)	-	5	76	75	94	52	27

Conditions:

Leach

Time : 15 min, no heating up time
Temperature : 95°C
Initial Solids Content : 72.6 g/L
Agitation : 420 rpm
Agitator : 13.2 cm ø, 6 bladed radial turbine
Solution Volume : 30 L
Lignosol : 0 g/L

Standard Flotation

Solids : 200 g
Volume : 1 L
Agitation : 1000 rpm
Roughing Time : 6 min
Total Time : 10 min

Test 11.1

Ferric Chloride Leach of Ronaldo Gubbins Lead Concentrate

	Element						
	Total	Pb	Zn	Fe	S ^{tot}	S [*]	Ag
<u>Feed</u>							
Assay (%)	-	51.5	6.6	6.5	18.9	-	0.1454
Mass (kg)	2.533	1.3	0.167	0.1606	0.479	-	3.68 g
<u>Residue</u>							
Assay (%)	-	3.9	12.3	14.8	43.7	21.1	0.126
Mass (kg)	0.962	0.38	0.12	0.14	0.42	0.20	1.21 g
Department (%)	-	3	71	86	88	42	33
<u>Flot Tails</u>							
Assay (%)	-	6.9	5.6	13.8	17.7	7.4	0.124
Mass (kg)	0.216	0.015	1.012	0.03	0.038	0.016	0.27 g
Department (%)	-	1	7	18	8	3	7
<u>Flot Conc</u>							
Assay (%)	-	3.3	14.4	15.2	51.9	25.0	0.1286
Mass (kg)	0.755	0.25	0.11	0.11	0.39	0.19	0.97 g
Department (%)	-	2	65	70	82	39	26

Conditions:

Leach

Time : 15 min, no heating up time
Temperature : 95°C
Initial Solids Content : 91.8 g/L
Agitation : 420 rpm
Agitator : 13.2 cm ø, 6 bladed radial turbine
Solution Volume : 30 L
Lignosol : 0 g/L

Standard Flotation

Solids : 200 g
Volume : 1 L
Agitation : 1000 rpm
Roughing Time : 10 min
Total Time : 18 min

Table 12a

**Summary of Sulphur Lixiviation From the Residues
Resulting From a Ferric Chloride Leach of Sullivan Lead Concentrate**

Test	Mass (g/L)		Residue Assay (g/kg)		Sulphur Leached		Sulphur Recovered From Solution			Comment
	Initial	Final	Initial	Final	(g/L)	(%)	Mass (g/L)	Assay (g/kg)	Recovery (%)	
12.1	200	164	261	8	50.9	97.5	31	999	61	lime leach
12.2	125	74	261	37	29.9	91.6	28	999	92	fresh carbitol
12.3	125	74	162	5	32.3	98.9	31	999	96	recycled carbitol
12.4	333	247	261	7	85.2	98.0	77	994	90	360 g/L (NH ₄) ₂ S
12.5	333	247	261	5	85.7	98.6	88	997	103	240 g/L (NH ₄) ₂ S
12.6	333	257	261	16	82.8	95.3	123	948	141	ammonium sulphite
12.7	333	260	306	15	98.0	96.2	140	995	143	(NH ₄) ₂ SO ₃ + H ₂ S purification
12.8	333	235	306				112			(NH ₄) ₂ SO ₃ +H ₂ S + H ₂ S purification

Table 12b

Semi-Quantitative Analysis of the Sulphur Products

Test	Analysis (g/t)																						Comment
	Al	Sb	As	Ba	Bi	Cd	Ca	Cr	Cu	Ge	Fe'	Pb	Mg	Mn	Mo	Ni	Si	Ag	Tl	Sn	Ti	Zn'	
12.1	50	10	-	7	0.5	0.4	1000	-	100	1	1000	70	100	-	20	5	400	30	0.4	4	20	1000	lime leach
12.3	7	1	-	2	-	0.5	7	-	3	-	200	20	20	2	-	-	200	1	-	0.1	0.7	400	recycled carbitol
12.5	2	400	30	-	-	0.5	0.4	0.5	400	-	30	20	7	2	-	-	20	70	0	3	-	300	ammonium sulphide
12.6	7	100	40	-	-	0.5	0.2	-	5	-	100	10	7	-	2	-	50	4	-	-	2	70	ammonium sulphite

* estimates

nb - detection limit for Te <10 g/t

Se is not analyzed for

Table 13

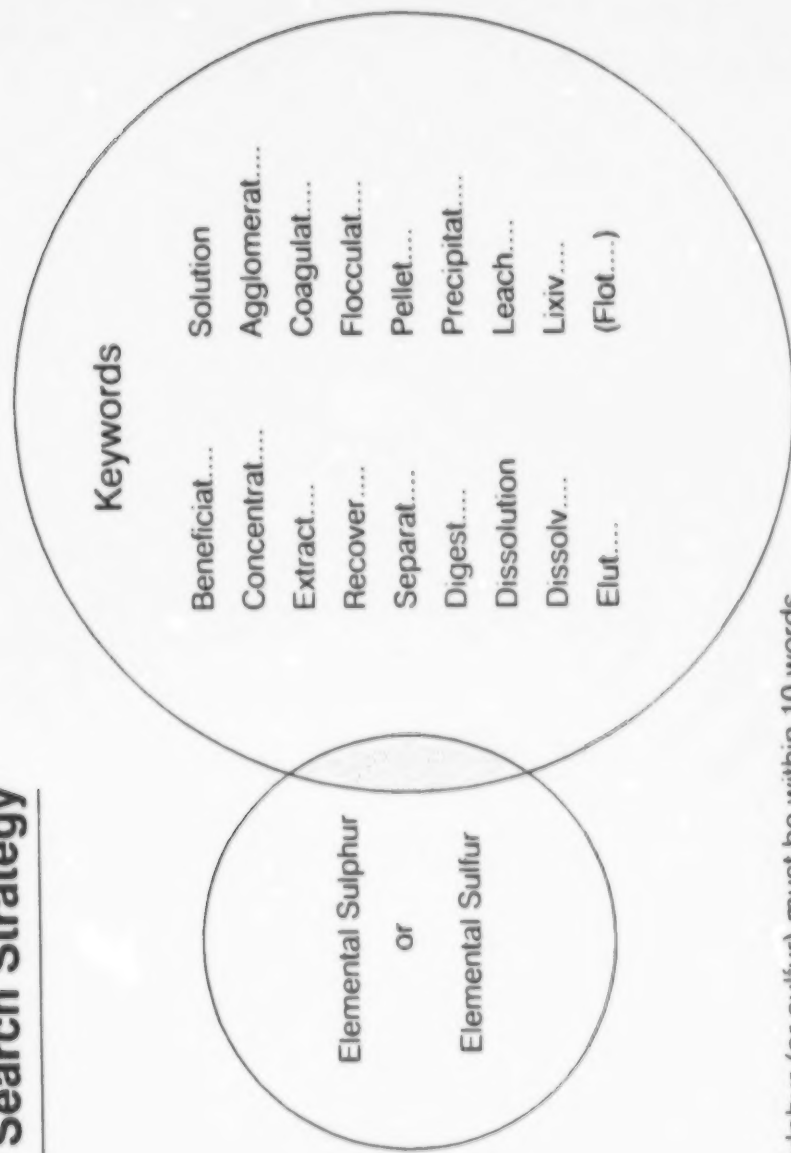
Hot Sulphur Filtration Residue Grinds

Grind	A	B	C	D	E	F	G	H	I	J
Micrometres	% in Size Fraction									
+147	1.9					24.1	1.7	26.9		
+104			0.03	0.02	0.01				11.5	12.5
+74 -147	18.4	9.7				27.7	23.3	21.1		
+74 -104			1.4	0.11	0.03				14.8	14.3
+53 -74	12.8	11.7	5.3	1.9	0.88	6.3	13.3	7.0	9.8	10.7
+43 -53	10.0	15.2	14.0	11.1	4.8	8.9	15.5	7.0	11.5	10.7
+38 -43	5.6	5.9	10.8	6.8	9.5	3.6	4.0	4.1	6.6	7.1
-38	51.3	54.7	68.6	80.0	84.8	29.5	42.2	33.9	45.9	44.6
Time/(min)	10	10	20	30	40	5	10	5	5	5

Mill - 25 cm ø x 35 cm rubber lined rod mill (RLRM #4)
 - + 30 kg steel rods
 - + 2 kg feed
 - + 1 L water

Fig 1

Literature Search Strategy



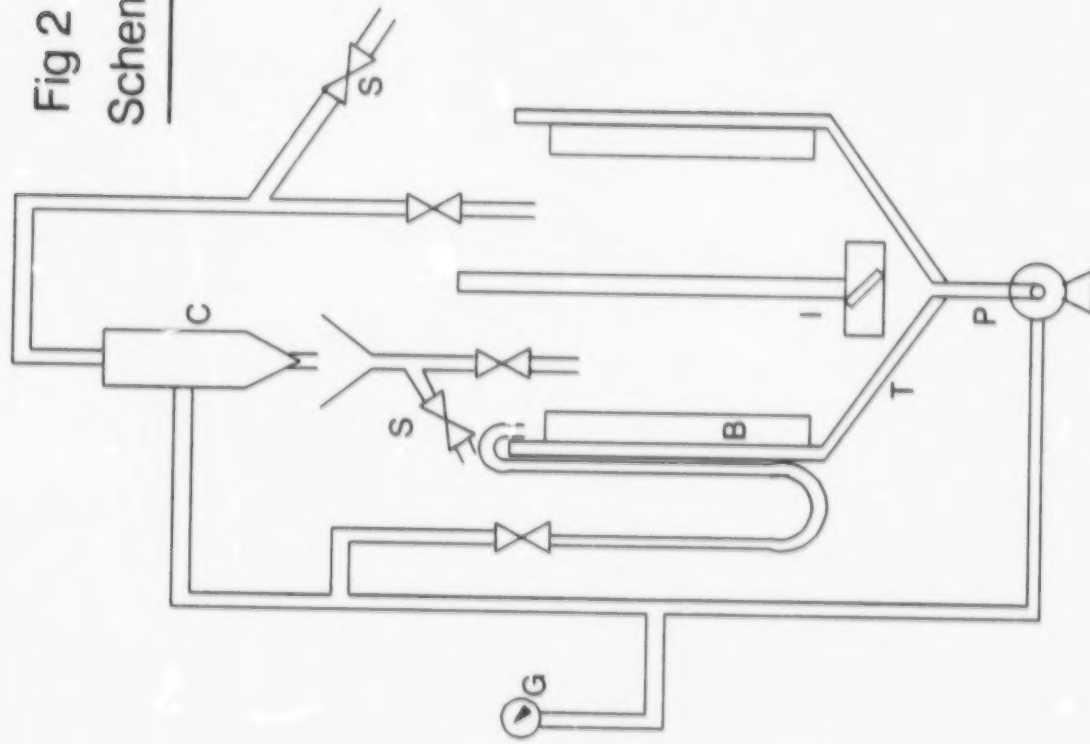
The term elemental sulphur (or sulfur) must be within 10 words of a keyword in the title or abstract.

date 3 Nov 1987 - 21 data bases
1064 references

of which about 200 were useful and not duplicated.

Fig 2

Schematic Diagram of Cycloning Apparatus



- T Tank
- B Baffles X4
- I Impellor
- C Cyclone (glass)
- P Peristaltic Pump
- G Pressure Gauge
- S Sample Points

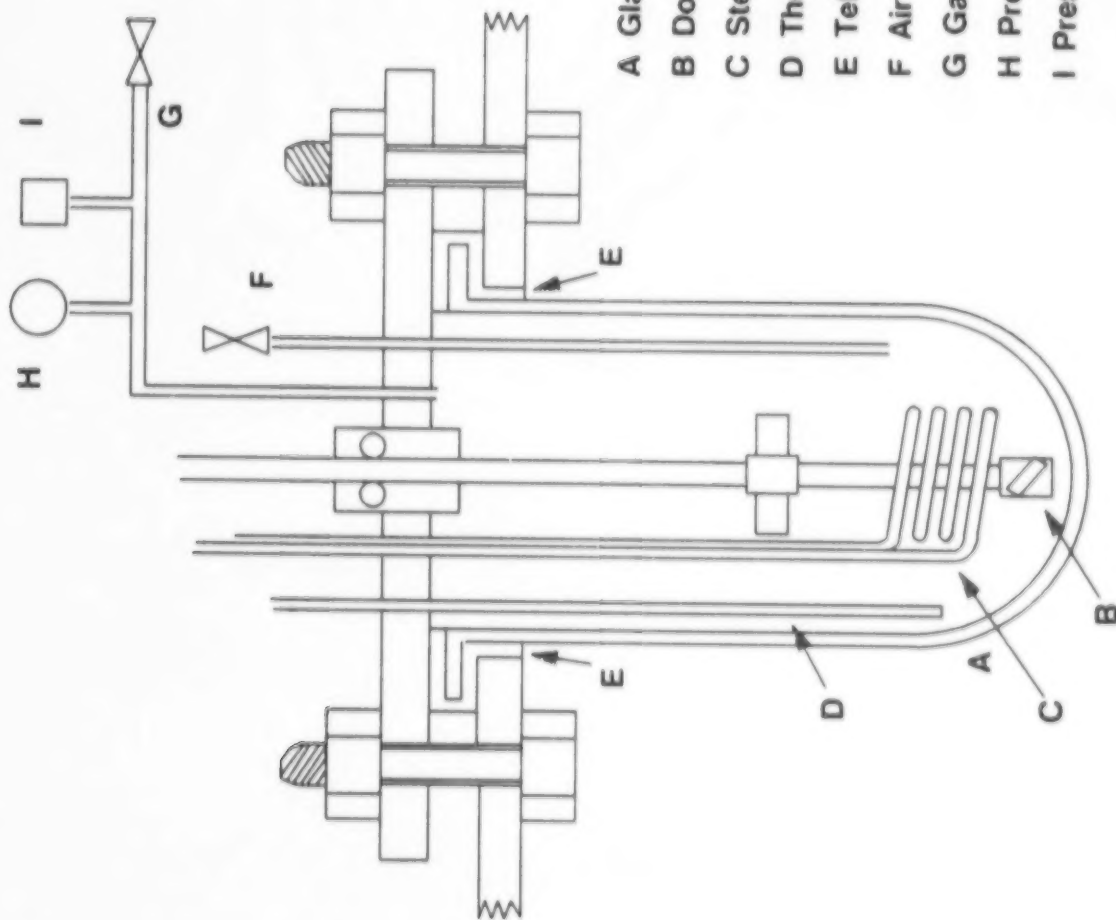


Fig 3
Schematic Diagram of
the Glass Autoclave

- A Glass Vessel
- B Double Bladed Agitator
- C Steam Coils
- D Thermocouple Well
- E Teflon Collar
- F Air Inlet
- G Gas Outlet
- H Pressure Gauge
- I Pressure Relief Valve

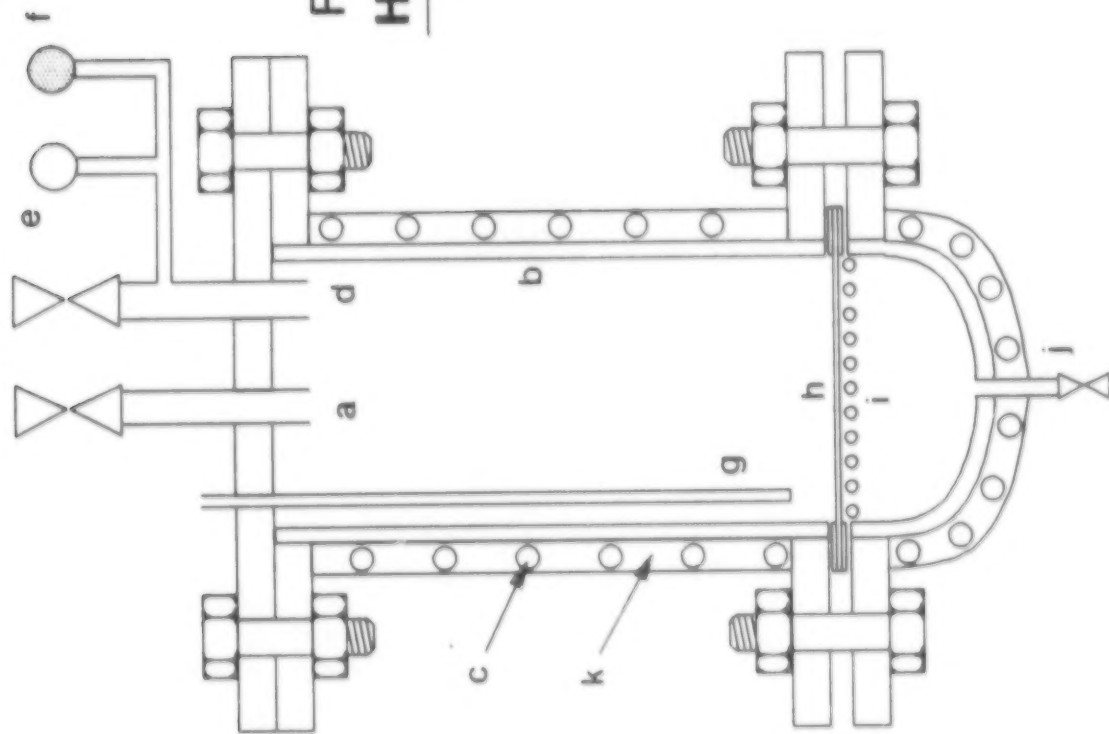


Fig 4

Hot Sulphur Filtration Apparatus

- a Feed Inlet
- b Steel Filter Wall
- c Copper Steam Coil
- e Pressure Gauge
- f Rupture Disc
- g Thermocouple Well
- h Gasketed Filter Cloth
- i Backing Screen
- j Clean Sulphur Outlet
- k Thermon Cement

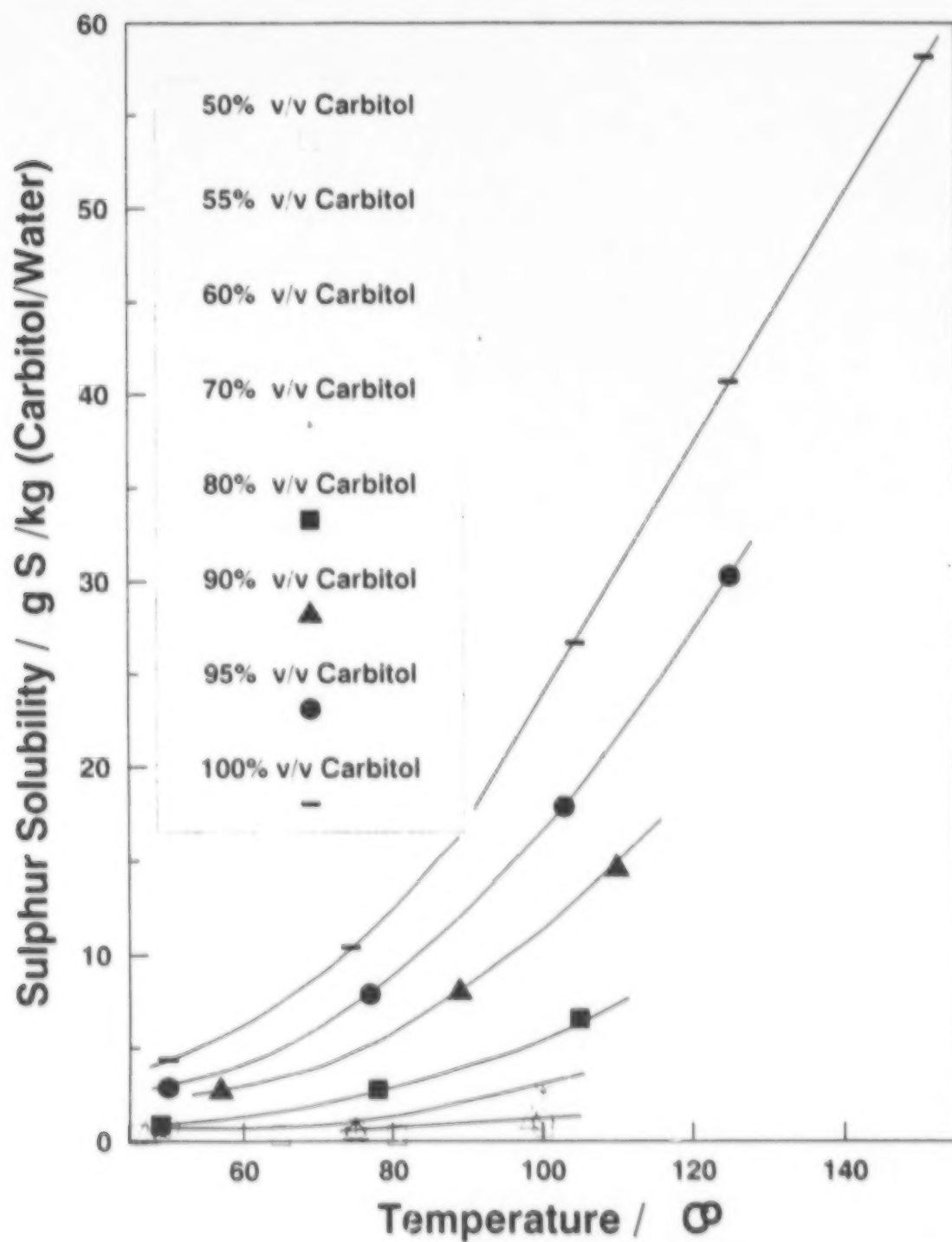


Fig 5 Sulphur Solubility in Carbitol

